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REFINER
AND NATURAL GASOLINE MANUFACTURER

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The Look Box

THE production of lighter fractions from gas in East Texas has developed into a gasoline plant practice of major importance, and a practice in which virtually all of the important manufacturers of gasoline in this rich gas area are engaging. To secure up to the minute information on this subject John C. Albright was assigned the task of interviewing those plant operators in the East Texas field that are operating newly installed fractionating equipment to secure data on the general scheme of gathering and transporting these light products, together with information dealing with individual plant practice, system hook-ups, and equipment employed. The full report is presented elsewhere in this issue.

This Month

Also, "The Constitution of Cracked and Un-Cracked Asphalts," a paper presented before the recent A.S.T.M. meeting, by E. S. Hillman and B. Barnett, is presented in this issue. The paper supplies chemical evidence supporting the conclusion drawn from the rheological properties of the cracked asphalts, that their greater degree of dispersion as compared to straight-run asphalt can be accounted for by the greater aromaticity of the maltenes, the authors state. It is shown that the components in low-level cracked asphalt have in all probability average molecular weights lower than the corresponding components in a straight-run residue. The authors then discuss another important point or two—but you take it from there.

J. A. Campbell does not like certain plant practices in gasoline plant operation and is especially displeased with the idea of throwing cold fully-condensed gasoline back into columns as reflux. Those who know Campbell personally will appreciate his informal style, those who have read others of his non-technical technical articles in THE REFINER during recent years will feel that an old friend is back, while those who meet him for the first time in this discussion of fractionation practices, like those just mentioned, will find something to think about, perhaps to advantage in his pointed remarks. Needless to state, they are his own.

E. C. McAninch of the Oklahoma Natural Gas Company recently contributed a discussion "Maintenance of Regulators" to The Oil Weekly, sister publication to The Refiner, and he did such a good job of it and presented his discussion so interestingly that the article is republished in this issue for the benefit of those thousands of readers in natural gasoline plants, refineries and stabilization plants who have to do with the operation of regulators, and the success of whose operations so frequently are dependent upon the performance of good regulators.

P. B. Shannon crowds a deal of interesting and up-to-the-minute information on the use of liquid-level indicator set-ups, speaking chiefly of manometers, in one discussion; his numerous illustrations aid in relating a long story in small

space and they will serve well as installation guides when so needed.

L. C. Trescott "started something" last issue with part one of his Taking Stock series. Part 2, and the conclusion, appears in this issue. In this part Trescott leaves materials behind and talks about the job and the man and tells management a few things also.

V. W. Garten, W. C. Moyer and H. T. Quigg by means of a little text and a large number of informative and valuable charts, in their discussion of the relationship between laboratory column distillations and A.S.T.M. end points, add much to an important investigation reported on in The Refiner in 1933.

L. H. Brandt in his timely and interesting discussion of the use of ammonia for corrosion control presents a deal of practical information along with technical data on the use of this widely used alkali.

Dog Days

AFTER Labor Day come the Dog Days. With the coming of Dog Days the season of high demand for motor fuels and motor lubricants is over. Such has been the condition in past years. Such will be the condition this year—a sharp and sudden decrease in demand. This is true because the schools are in session, the vacation period has passed, cooler or inclement weather prevails and the motorist does not drive as he does during the warm months.

The refining industry must take this condition into consideration again this year as in past years. The industry must prepare for the decrease in motor fuel demand. In the production branch of the industry it should be borne in mind that refineries can not use the excessive quantities of crude that is required in the spring and summer. Production can not go ahead full blast and continue to increase crude oil stocks without danger of such an increase softening the market during the fall or winter. When crude prices lower because of an over abundance of production, refinery products generally follow; dropping to lower levels. Lower levels than those prevailing so far throughout this year will endanger over-all profits for the industry.

The Dog Days are curtailing days; days for adjustment of operations and close study of the statistical and economic conditions. Likewise they are excellent days for a general survey of the plant processes, equipments and material stocks; and a good time for repairs and general overhauling in preparation for the coming winter months.

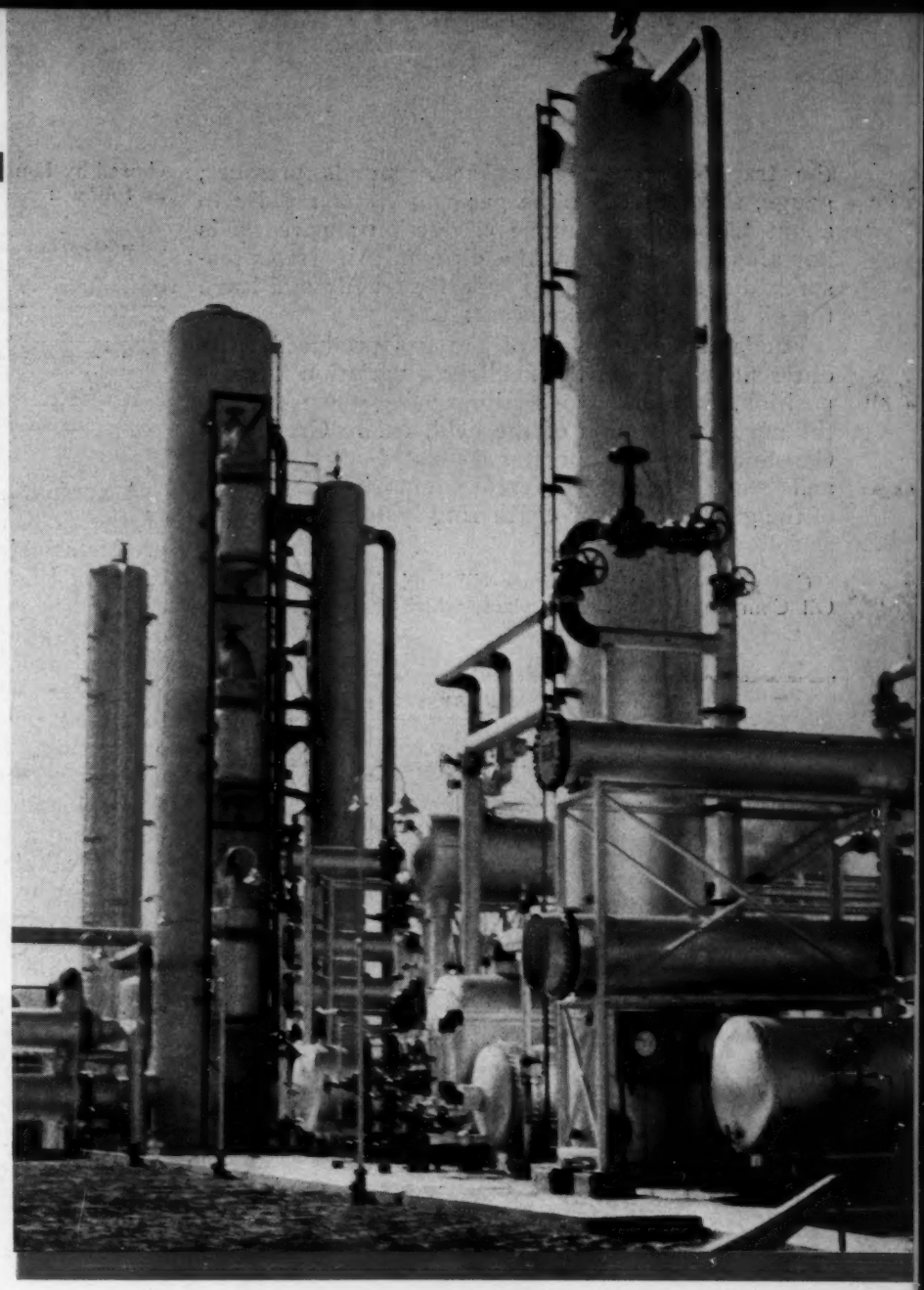
Michigan Crudes

THE growing importance of Michigan as an oil-producing state has prompted the United States Bureau of Mines, Department of the Interior, to extend its study of the general characteristics of crude oils to include representative samples from 13 fields in 8 counties in the lower peninsula. The resulting report indicates that the oils are predominantly paraffin-intermediate-base, wax-bearing crudes, yielding 25 to 35 percent of highly paraffinic gasoline and about the same amount of kerosene and gas-oil combined, 12 to 15 percent of lubricating oil stock, and 20 percent of residuum with a carbon residue of about 6 to 7 percent, according to the Bureau of Mines method of crude-oil analysis and classification. There appears, also, to be some correlation which has been portrayed graphically, between certain properties of the crude oils and both the geographical location and geological structure of the oil-producing areas within the state. The producing horizons from which the samples were obtained are represented by the Berea sandstone, Upper Traverse and Traverse limestones, and the Dundee and Trenton limestone formations.

The samples from the Dundee limestone are similar to each other with certain exceptions. These differences appear in production from this formation outside of what usually is termed the Central Michigan area. The properties of certain crude oils produced within the area are much alike but show variations depending upon the distance of the producing pool from the center of the basin. The Traverse limestone samples show local variation but in general are not materially different from the Dundee production. The samples from the Muskegon and West Branch fields show considerable variation from the other limestone production in a number of properties. The Berea sandstone crude oil is characterized by relatively high viscosity of the vacuum fractions.

The 15 samples of Michigan crude oils, for which individual detailed analyses are given, were collected during May and June, 1936, and therefore represent current production, and the analyses are on a comparable basis. The oldest well covered by the samples was completed during November, 1927, and six of the samples were from wells completed in 1936. Analyses of a sample from the Bradford Third sand of the Bradford field, McKean County, Pa., is included for comparison.

Copies of Report of Investigations 3346, Analyses of Crude Oils From Some Fields of Michigan, may be obtained without charge upon request to the Information Division, Bureau of Mines, Washington, D. C.



Hanlon Gasoline Company recently installed new distillation and fractionation equipment for preparation of light fractions. The unit here shown is four miles northeast of Gladewater, in the East Texas field.

Production of Lighter Fractions *From Gas* In East Texas

J. C. ALBRIGHT

WITH a reported flowing life of about five more years, as estimated by competent oil producers, East Texas is destined to produce large quantities of both natural gasoline and butane. The average gas production over the field is about 240 cubic feet per barrel of oil produced, which, when processed will make an average of about 3.75 gallons per thousand cubic feet. However, when computing the amount of gasoline which the East Texas field can produce, it must be considered that the available gasoline varies according to the field separator pressures. For example, one manufacturer has compiled a graph which shows the result of many tests made in the field under varying trap pressures, and shows that when field conditions require a separator pressure of about 20 pounds, gauge, the gasoline production, based on a grade of 26/70, will be about 1.50 gallons per thousand. On the other hand, when

the traps are operated at zero pounds pressure, gauge, the content of the gas will climb rapidly to about 4.50 gallons. Butane production apparently is not affected greatly by the difference in trap pressures, due to its ability to vaporize readily and leave the oil under those conditions.

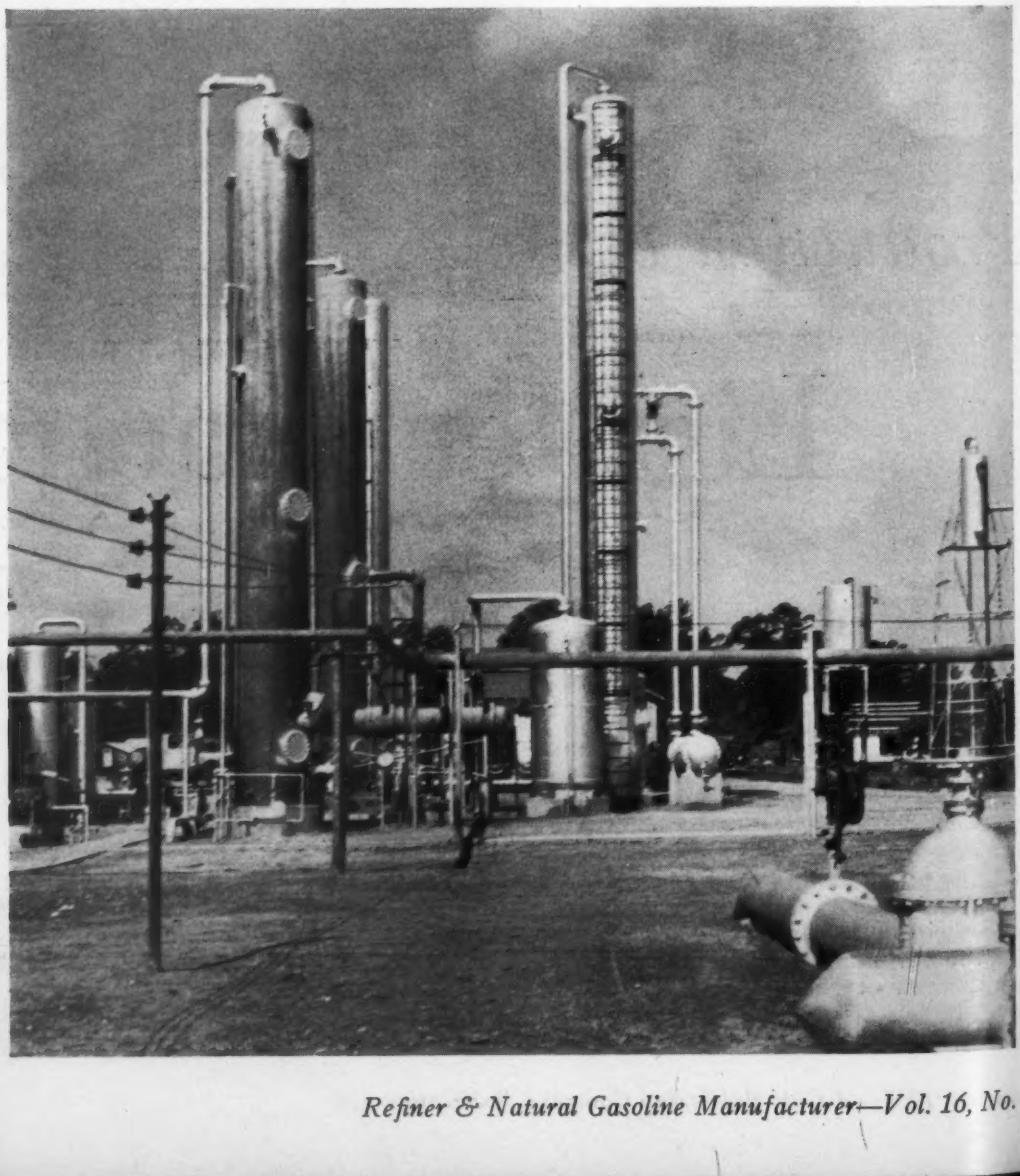
Fundamental analysis of the gas produced from different sections of the field show variation in composition, with no two sections being the same. In the northeast section of the field, where Gregg-Tex Gasoline Company operates a plant, both the "east" and "west" gas has a different composition. Analysis of the gas from the two gathering systems ars shown as follows:

Gas processed at the former Gilliland-General American Oil Company's Gladewater plant, which was recently purchased by Humble Oil & Refining Company, has an analysis as follows:

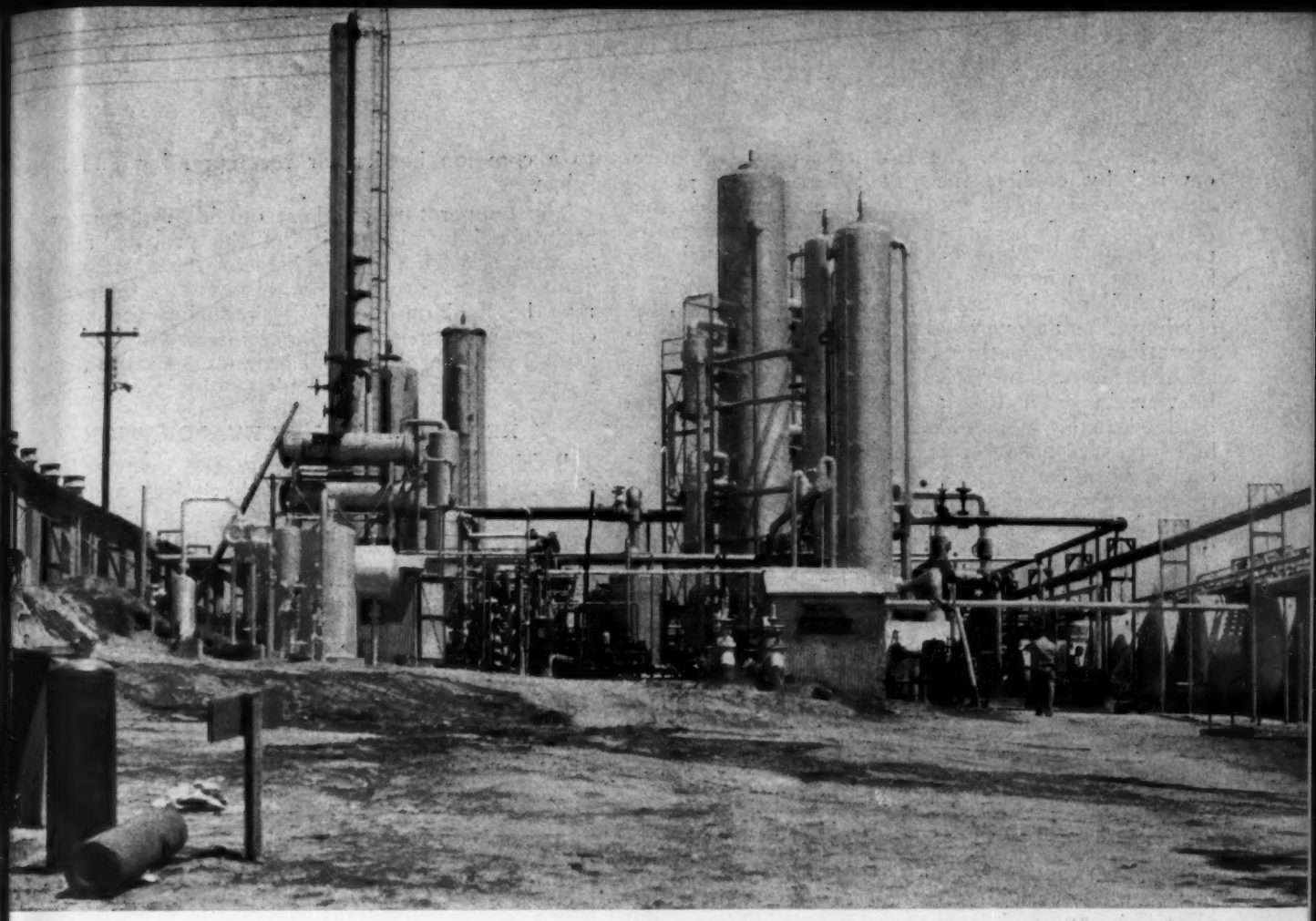
COMPOUND	EAST SYSTEM		WEST SYSTEM	
	Volume Percent	G.P.M.	Volume Percent	G.P.M.
Methane and air	40.47	.....	40.57	.....
Ethane	15.06	.....	15.90	.....
Propane	23.36	6.38	24.62	6.72
i-Butane	3.72	1.20	3.20	1.03
n-Butane	10.55	3.31	9.44	2.96
i-Pentane	2.11	.77	2.03	.74
n-Pentane	2.29	2.29	2.10	2.10
Hexanes and heavier	2.44	1.05	2.14	.92
Liquid volume at 60° F.	.....	3.89	.....	3.89

COMPOUND	Volume Percent	G.P.M.
Methane	44.13	.....
Ethane	9.50	.....
Propane	27.37	7.47
i-Butane	6.15	2.00
n-Butane	8.38	3.00
Pentane plus	4.47	3.70

A comparison between the two different sections of the field will show a slight difference in total products which may easily be liquefied, but the average of i-butane and heavier will show that about 8.7 gallons per thousand cubic feet are available for recovery, assuming that a given plant is operating at 100 percent efficiency. Not all of these hydrocarbons have been desired until comparatively recently, due to the absence of a market sufficient to absorb total production. Since industrial plants, communities without access to natural gasoline, and refiners with thermal polymerization plants have gradually been increasing a market for the lighter fractions, particularly the butanes, East Texas manufacturers are in a position to furnish large quantities of these products for several years to come.



Lisbon Gasoline Company's plant in Rusk County in the East Texas field. In the center of this picture is shown the intercooler on the side of the high-pressure fractionating column.



Parade Gasoline Company's Joiner area, Rusk County, East Texas field gasoline plant, showing the outside propane coolers on the absorber.

BUTANES AND PROPANES

Facilities have been, and are being installed in most of the gasoline plants in East Texas to recover all of the butanes and some of the propanes, if desirable. A market for the butanes, especially, is being worked out by Humble Oil & Refining Company at Baytown, Texas, where this company is completing a large thermal polymerization unit which will require a large portion of the available butanes in the gas produced in East Texas. Early in the spring of 1936 this company converted one of its 8-inch oil lines leading from the East Texas field to Baytown for transmission of natural gasoline and butane. Hanlon-Buchanan, Inc., formed the gathering pipe line system to deliver gasoline from many of the plants to this line at London, where Humble Oil & Refining Company has a large plant of its own.

In the system of pipe lines, the plants having connections will manufacture gasoline of one grade, containing all of the butanes that can be handled and the raw gasoline will be fractionated, or stabilized to the desired grade. The overhead, principally butane, will be retained by Humble Oil & Refining Company and processed for polymerization. Manufacturers not in the gathering system agreement will, perhaps, manufacture butanes in addition to the usual product, but will fractionate them from the gasoline in the field and ship separately.

Arkansas Fuel Oil Company installed one of the first gasoline plants to be built in East Texas, most of which were in use in other fields, in Louisiana and Arkansas, but were dismantled when gas volumes dropped so low that economical operation became impossible. Recently this company has added to its facilities so that the lighter fractions can be recovered, using high-pressure absorption for this purpose. Columns have been erected that will make it possible for any cut to be made from the raw gasoline, including propane, butane and low-vapor pressure natural gasoline. The fractionation equipment installed by Arkansas Fuel Oil Company may be used to manufacture butane for the refinery at Shreveport, when and if that company builds a "poly" plant.

Gregg-Tex Gasoline Company, Hanlon Gasoline Corporation, Sabine Valley Gasoline Company, and Parade Gasoline Company are all connected to the pipe line gathering system and make all shipments regularly through Humble Pipe Line Company's line to the Gulf Coast. Facilities have been installed in these plants which will permit the maximum extraction of all the desirable hydrocarbons contained in the gas, especially the butanes and heavier. Humble Oil & Refining Company, being one of the interested parties in the production of "poly feed", has also installed similar equipment for extracting this material. Sinclair Prairie Oil Company has two plants in the field, but ships the usual grades of stabilized natural, fractionated to requirements in the field. Lisbon Gasoline Company employs compression and absorption to extract the gasoline from the gas passed through its two plants, and fractionates commercial gasoline in the field, with butanes shipped by tank car and tank truck.

Both of the plants operated by Lisbon Gasoline Company, the main unit at Moore and the Grisson plant, are operated in conjunction with each other. The Grisson plant is more of a field unit than a complete gasoline plant, as all oil is distilled at the Moore plant that passes through the high-pressure

absorbers. Likewise, the compression gasoline recovered by cooling the gas before it enters the absorbers is shipped to the Moore plant and fractionated into the desired cuts. The entire volume of raw gasoline is pumped continuously to a primary fractionator where the 26/70 gasoline is taken from the reboiler, with butane, isobutane and propane condensed as an overhead product. This company is operating fractionating columns to cut practically all of the iso-butane from the remaining butane and heavier, in order that a larger amount of normal butane can be included in the stable product. The high-pressure absorber is equipped with an outside cooler, with connections at about half way up the column where the oil is drawn from the tower. It flows downward to an inclosed tubular unit and returns to the plate next below where it was withdrawn. The overhead material separated from the stabilizer charge is fed to a second column where the propane-free butane is manufactured as a bottom product, and butane-free propane comes off overhead. Market requirements determine the mode of operation when separating the two hydrocarbons, and propane may be included in the base product if desired. Likewise, if markets require a product with a preponderance of propane, butane can be withdrawn for the overhead connections and liquefied.

OPERATION OF PLANTS

The operation of the Gregg-Tex Gasoline Company, Parade Gasoline Company, Sabine Valley Gasoline Company and some of the units of Humble Oil & Refining Company are more or less similar to the methods used by Hanlon Gasoline Corporation. Low-pressure absorption is employed at these plants, but including two-stage contact between the gas and the absorption oils. The first stage absorber employs propane refrigeration to maintain a relatively low temperature on the oil as it flows downward through the column. Large size columns were installed running to 7 and 8 feet in diameter and ranging from 55 to 62 feet in height. Propane fractionated from the gross production when depropanizing the "butane plus" production is liquefied and used in a continuous recirculatory system. Several compressors, driven by the usual direct-connected gas engines, are used in each of these plants as "propane" machines which are operated at sufficient pressure to liquefy the propane in the system at normal condensing temperatures.

The propane is received in the usual type of accumulator drum at the outlet of the condensing sections. Liquid-level controls determine the volume of liquid leaving the accumulator drum, as it flows through piping to its place in the refrigeration system. An auxiliary tower is erected beside the refrigeration-operated absorber as a propane tower, where the liquid enters at the top connection. Several plates are built into the column for distribution of the liquid as it flows out to the refrigerators attached to the sides of the low temperature absorber. Each of these refrigerator absorbers is equipped with six coolers, placed in regular order from the top to the base, and divided equally on each side. Six units are included in the cooling system, staggered from side to side to permit equal draw-off of the absorption oil as it flows down the column. Vapor lines reconnect the coolers to the top of the propane tower so that vaporized propane may return to a common header for recompression and liquefying.

The temperature on these outside refrigerators is maintained by the purity of the propane and the amount of back pressure held on the system. With commercially-pure propane, almost any temperature may be held on the oil by reducing the pressure on the system at the auxiliary tower. However, with a mixture of propane and butane, desirable temperatures are somewhat difficult to obtain.

REFRIGERATION BY EVAPORATION

Refrigeration is obtained by the evaporation of the propane liquid in the outside coolers, and not by direct expansion as with some systems that employ expansion valves at the cooler sections. After the propane has become vaporized, it passes from the header to a supplementary gas cooler near the absorber so that all the benefit of the temperature may be enjoyed, and to prevent the possibility of slugs of liquid pulling over into the compressor cylinders of the propane machines.

Make-up of the propane liquid is taken from the reflux condenser at the depropanizing column in all of these plants, but where special equipment is not available, debutanizing the propane is not feasible, and when the capacity of the gasoline transmission line has been met, obviously some of the butane must be removed from the raw gasoline in the depropanizing columns. When this occurs, contamination of the refrigerant causes higher temperatures to occur within the outside coolers than when a pure propane is used.

The absorption oil used in these refrigerator absorbers is what is termed in the field as "self-building". The heavier fractions in the gas are liquefied in the beginning and continue as the plant is operated. With a rich gas volume of approximately 15,000,000 cubic feet processed each day, the circulation of this oil is about 320 gallons per minute, obtained with motor-driven, or steam turbine-driven centrifugal pumps. The gravity of this "self-building" absorption oil is about 60° A.P.I., has a Reid vapor pressure of about 1½ pounds, an initial boiling point of 150°F., and an end point of about 375° F., A.S.T.M.

Two separate distillation units are used for this type of absorption plant, one for this light self-building oil and the other for the usual type of absorption oil. As the gas is passed through the refrigeration unit, it is received by a scrubber and immediately enters the secondary column where it is contacted with about 184 gallons per minute of regular absorption oil. The pressures on these units vary, but the usual procedure is to admit the cooled raw gas to the primary absorber at a pressure of about 55 pounds, gauge.

GAS CONTRACTS

The activities of the gasoline manufacturers in the East Texas field, and elsewhere, have caused some of the producers of oil and gas to demand a change in the existing casinghead gas contract, adopted by the Natural Gasoline Association of America, on November 7, 1932. In making this demand, the companies have observed that paragraph 1 of the contract specifies the purpose of conveying the casinghead gas from the seller to the buyer, and says:

"The gas hereby sold is conveyed to the buyer

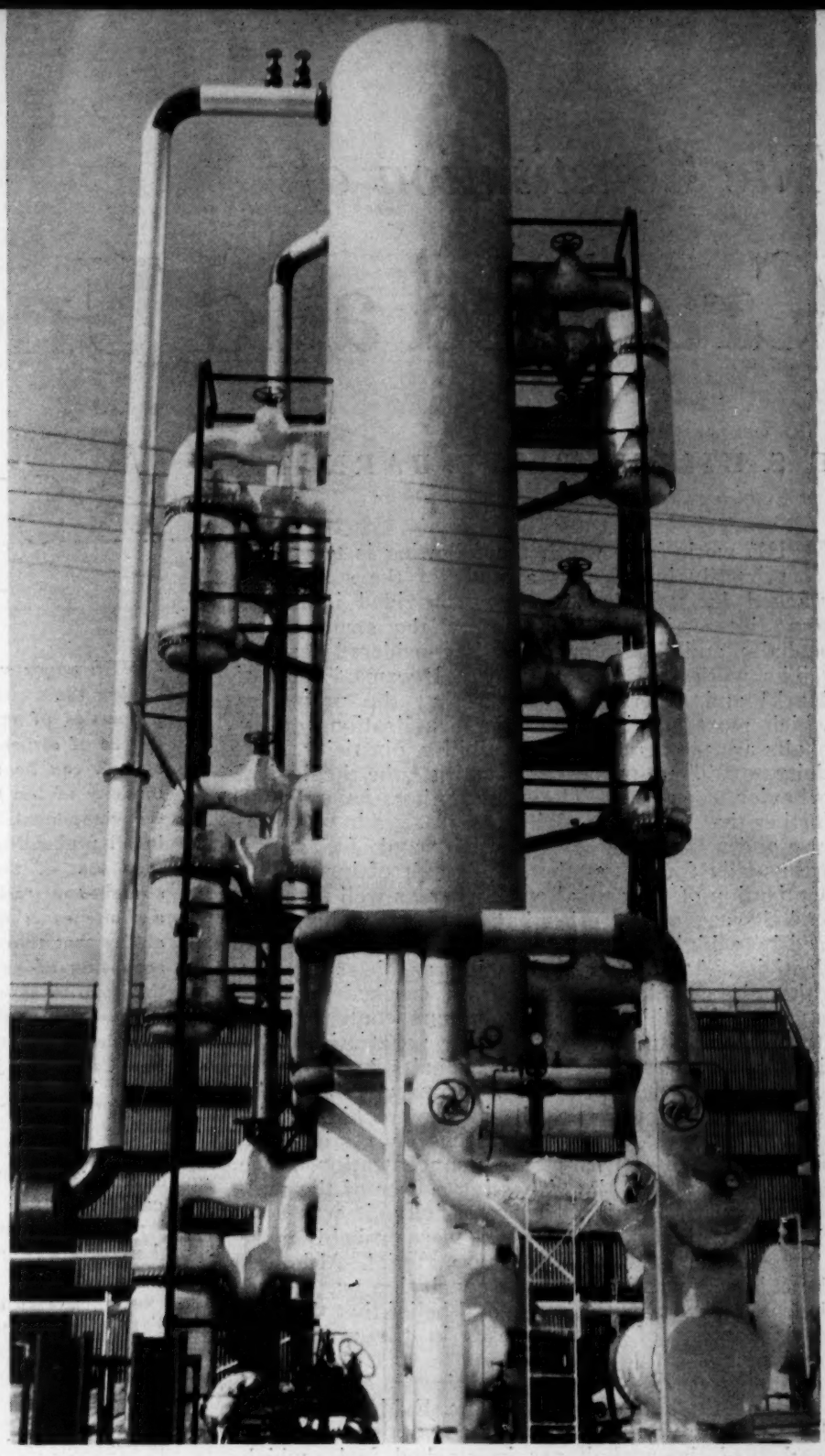
for the purpose of manufacturing therefrom gasoline or such other product or prducts as may be manufactured at buyers plant."

While the producers of gas and oil are not making objections particularly because of the plant operator manufacturing these "other products," they want paragraph 10 of the casinghead contract changed, or supplemented to include such revisions as asked for by Amerada Petroleum Corporation, for example, which concern has drawn up an agreement which reads:

"10.A — Additional Products. Buyer desires to extract, condense and save certain additional petroleum products, i.e., butane and propane, such operation being hereby specifically authorized, and for the purpose of more efficiently performing such operation, buyer is now or may install additional equipment at its plant. Upon beginning such operation and continuing during the period or periods such products are saved and sold, buyer shall, in lieu of all obligations, if any, imposed by the other provisions of this contract with reference to said additional products, compensate seller for the rights herein granted by paying seller, in addition to the sums of money otherwise provided to be paid under this contract, the following sums computed at a price per thousand cubic feet for casinghead gas delivered hereunder, to-wit:

"(a) When buyer's actual average gross sales of butane is two (2c) cents or less per gallon for the period which is being accounted for, fifteen (15%) percent of the gross sales price of said additional products actually saved and sold from buyer's plant, attributable to casinghead gas delivered by seller to buyer under this contract."

The amendment to the casinghead contract goes on to state that 20 percent of the gross sales price shall be remitted to the seller if the butane is selling between 2 and 4 cents per gallon; if between 4 and 6 cents, 25 percent shall be paid, and if 6 cents or more is received from the sale of butane, the seller shall receive 33 1/3 percent of the gross sales receipts.



View showing details of installation of outside propane-cooled absorber with units mounted on the side of the column. Hanlon Gasoline Company, Gladewater, Texas.

This all goes to indicate that the business of fractionating butane and other products from casinghead gas, in addition to the usual grade of 26/70 gasoline is becoming more and more of a major enterprise, and only needs the perfection of small, low cost "poly" plants to provide an outlet for practically all of the butanes available in casinghead gas.

East Texas as a field, is pretty well saturated with gasoline plants, with all leases contracted for by operating companies, who need only a definite outlet to enable them to produce large quantities of low-boiling petroleum fractions for many years to come.

The Constitution of

Cracked and Uncracked Asphalts

E. S. HILLMAN[1] and B. BARNETT[1]

THIS paper supplies chemical evidence supporting the conclusion drawn from the rheological properties of cracked asphalts, that their greater degree of dispersion as compared with straight-run asphalt can be accounted for by the greater aromaticity of the maltenes. It is further shown that the components in low-level cracked asphalt have in all probability average molecular weights lower than that of the corresponding components in a straight-run residue. The chemical structure of asphaltenes is discussed, together with the probability that they consist of a combination of units consisting of cyclic groups of probably no more than two or three rings condensed together.

Presented at the Fortieth Annual Meeting of the American Society for Testing Materials, held at New York City, June 28-July 2, 1937.

THE work from which the following is taken was primarily intended as a study of the stability of cracked fuels. It has however, provided indirectly some interesting information on the structure of asphalts, and this information is considered here.

The work of Pfeiffer and van Doormaal[2] and of Mack[3] and others has shown that the properties which most affect the practical applications of asphalts are temperature susceptibility, plasticity, elasticity, etc. These properties constitute the rheological behavior of asphalts. The same authors have shown further that low susceptibility to changes in temperature and a high degree of plasticity and of elasticity are usually found together, the result of incomplete peptization of the asphaltenes, whereas well peptized asphalts are, on the other hand, generally characterized by a high susceptibility with less plasticity and elasticity. At the same time it was observed that although asphalts prepared from cracked residues have considerably higher asphaltene contents than straight-run bitumens of the same penetration from the same crude, the cracked asphalts have higher temperature susceptibilities. Consequently, the cracked asphaltenes must be in a more highly dispersed state than the asphaltenes in straight-run bitumens despite their greater concentration. Since it was known that the dispersing power of the maltenes increases with their aromaticity, it was concluded that the cracked maltenes were probably more aromatic than the straight-run material. Although the evidence was indirect, the conclusion was nevertheless in accord with the general belief that the aromatic content of oils increases in the process of cracking.

There were, however, difficulties in the way of accepting the above point of view. Evidence against this theory is the instability or apparently poor dispersion of the asphaltenes in cracked fuels, which manifests itself in the well-known sludge deposits that such fuels were once so prone to produce.

However, this poor dispersion is not an unavoidable property of the system. One of the authors had found in 1926 that a characteristic of the high-level residues produced at that time, with their high content of poorly peptized material, was the low proportion of maltene resins and that by extracting them from a large quantity of the residue and mixing back with proportionately less residue, cracked asphalts were obtainable of superior ductility, gloss, etc., indicating a high degree of dispersion. Further, attempts to recover the components from such resin-enriched mixtures by extracting with solvents failed; the carbenes and carboids, always present in high-level residues, could no longer be detected, by the usual methods then existing. In other words, the mutual solubility or dispersibility of the components was such that normal extraction methods failed to separate them, and a separation could be effected only by an elaborate extraction with a carefully graded series of solvents. This was definite evidence that the material comprising what is usually considered the heaviest and most intractable components of the cracked residue could be adequately dispersed.

NATURE OF THE MALTENES

From the observation given in the previous section it was evident that incorrect results would be obtained if the proper solvents were not used for the separation of the components. For this purpose a series of solvents of increasing internal pressure was chosen as follows, in the order shown:

Isopentane
Hexane
Carbon tetrachloride
Benzene
Carbon bisulfide

To these was added pyridine which, owing to its polar nature, is a better solvent for many compounds than even carbon bisulfide.

Theoretically the series should start with ethane

[1] Research Chemist, Shell Development Co., Emeryville, Calif.
[2] J. P. Pfeiffer and P. M. van Doormaal, "The Rheological Properties of Asphaltic Bitumens," *Journal*, Inst. Petroleum Technologists, Vol. 22, No. 152, June, 1936, pp. 414-440; *Kolloid-Zeitschrift*, Band 76, Heft 1, pp. 95-111 (1936).
[3] C. Mack, *Proceedings*, Assn. Asphalt Paving Technologists, Technical Sessions held at Washington, December 9, 1933, p. 40.

TABLE 1

Analyses of Dubbs Residue Fractions

Viscosity Saybolt Furol 210 F. = 27.8 sec.

FRACTION		Internal Pressure of Solvent, Atmos. per Sq. Cm.	Percentage of Original Residue	CHEMICAL ANALYSES, PERCENT						Carbon-Hydrogen Ratio	Density 20 C.	Average Molecular Weight
				Carbon	Hydrogen	Nitrogen	Sulfur	Ash	Oxygen			
Isopentane soluble	Cut No. 10	....	34.17	87.95	10.60	0.3 (5)	0.7 (0)	Trace	0.40	8.3	0.9681	270‡
	Cut No. 9	1180	11.88	87.90	10.00	0.5 (5)	0.8 (1)	Trace	0.74	8.8	0.9975	345‡
	Cut No. 8	1200	4.06	88.65	9.85	0.7 (4)	0.8 (5)	Trace	0.00	9.0	1.0136	344‡
	.....	....	....	.....	.....	.....	.....	.....	....	....	.....	....
	Cut No. 7	1250	7.09	88.20	9.70	0.7 (7)	0.8 (8)	Trace	0.45	9.1	1.0220	350‡
	Cut No. 6	1330	4.39	88.35	9.40	0.9 (7)	0.9 (1)	Trace	0.37	9.4	1.0403	353
	Cut No. 5*	1420	7.36	88.45	9.20	0.9 (8)	0.8 (3)	0.00 (8)	0.53	9.6	1.0453	374‡
	Cut No. 4*	1520	4.15	88.35	8.85	1.0 (2)	0.7 (4)	0.01 (3)	1.03	10.0	1.0583	384‡
	Cut No. 3*	1640	5.76	88.50	8.75	1.1 (1)	0.8 (3)	0.01 (6)	0.79	10.1	1.0683	395‡
	Cut No. 2*	1800	3.62	88.25	8.40	1.2 (4)	0.9 (5)	0.06	1.10	10.5	1.0860	439‡
	Cut No. 1*	1900	2.52	88.05	8.10	1.3 (7)	0.9 (8)	0.30	1.20	10.9	1.1102	483‡
Isopentane insoluble	Hexane-soluble†	....	4.2	88.1	7.4	1.8	1.3 (3)	0.07	1.30	11.9	1.1-1.2	490§
	CCl_4-soluble‡	....	6.3	83.9	6.2	2.2	0.3 (7)	0.10	6.73	13.5	1.18	830§–870‡
	Benzene-soluble	....	2.0	86.8	6.1	2.3	0.8 (8)	0.17	3.75	14.2	1.19	880‡
	CS_2-soluble§	....	1.0	87.1	6.0	2.1	0.9 (0)	0.25	3.68	14.5	.....	817‡
	Pyridine-soluble‡	....	2.0	83.7	5.6	3.2	0.9 (5)	0.22	6.31	15.1	1.24	860‡

* Semi-solids at room temperature.
† Solid at room temperature. Melts 90 to 100 C.
‡ Powdery solids; the CCl_4 solubles melt at high temperatures with decomposition and swelling; the benzene-solubles also decompose, although with less swelling; and the heavy carbenes and carboids neither melt nor swell.
‡ In benzene, at the boiling point and freezing point.
§ In benzene, at the boiling point; and in naphthalene, at the freezing point.
‡ In naphthalene, at the freezing point.

or propane, but it was found more convenient to make a rough separation with isopentane; and then, when desired, to divide the isopentane-soluble part into fractions by dissolving in propane and successively adding increments of ethane or methane, removing the precipitate after each addition.

Ultimate analyses and also average molecular weights of fractions obtained in this manner are given in Tables I and II.

In Table I, the fractions were obtained from a Dubbs low-level residue from a Californian asphalt base crude. With the exception of the values for the average molecular weights, of which the plot flattens out or may even attain a maximum in the direction of the carboids, it will be seen that there is a regular gradation in the data given, showing the reality of the fractionation achieved. This will be dealth with later.

These results should be compared with those given in Table II for fractions obtained from a straight-run residue of approximately the same viscosity from the same crude. Below the dotted line in each table is the material that would be present in a 65 penetration asphalt for both the cracked and the uncracked residue.

The internal pressure of the solvent corresponding to the precipitate produced gives a basis for comparison of the fractions.

The greater aromaticity of the cracked maltenes is apparent from the fact that the specific gravity is higher for the same molecular weights, and that precipitates of the same molecular weight are ob-

TABLE 2

Analyses of Straight Run Residue Fractions

Viscosity Saybolt Furol 210 F. = 23.2 sec.

FRACTION		Internal Pressure of Solvent, Atmos. per Sq. Cm.	Percentage of Original Residue	CHEMICAL ANALYSES, PERCENT						Carbon-Hydrogen Ratio	Density 20 C.	Average Molecular Weight
				Carbon	Hydrogen	Nitrogen	Sulfur	Ash	Oxygen			
Isopentane soluble	Cut No. 19	....	9.51	86.6	12.7	0.10	0.49	0.01	0.10	6.83	0.9075	293†
	Cut No. 18	800	4.28	86.8	12.4	0.18	0.56	0.02	0.04	7.0	0.9292	373†
	Cut No. 17	845	0.48	86.7	12.4	0.18	0.69	0.02	0.01	7.0	0.9393	365†
	Cut No. 16	855	6.37	86.6	12.3	0.16	0.67	< 0.01	0.26	7.05	0.9340	391†
	Cut No. 15	915	1.71	86.3	12.0	0.28	0.56	0.009	0.85	7.19	0.9440	421†
	Cut No. 14	935	4.19	86.7	12.0	0.24	0.70	< 0.01	0.35	7.23	0.9460	413†
	Cut No. 13	975	3.04	86.9	12.0	0.34	0.75	0.02	....	7.22	0.9493	427†
	Cut No. 12	1010	3.61	86.4	11.8	0.32	0.81	0.03	0.64	7.3	0.9529	438†
	Cut No. 11	1035	2.19	86.4	11.7	0.36	0.79	0.01	0.74	7.36	0.9593	448†
	.....	....	....	.....	.....	.....	.....	.....	.....	.....	.....	....
	Cut No. 10	1055	5.80	86.3	11.5	0.43	0.66	0.02	1.01	7.51	0.9613	448†
	Cut No. 9	1090	5.80	86.5	11.4	0.53	0.79	0.01	0.77	7.58	0.9690	497†
	Cut No. 8	1130	6.84	86.8	11.3	0.56	0.92	< 0.01	0.41	7.68	0.9790	489†
	Cut No. 7	1195	3.61	86.8	11.0	0.77	1.15	< 0.01	0.27	7.9	0.9934	397†
	Cut No. 6	1235	4.56	86.6	10.8	0.87	0.97	0.05	0.71	8.02	1.0031	520†
	Cut No. 5	1290	2.18	86.8	10.5	0.99	0.97	0.04	0.70	8.26	1.0157	539†
	Cut No. 4	1330	1.90	86.6	10.3	1.08	0.87	0.05	1.10	8.41	1.0234	524†
	Cut No. 3	1360	1.33	86.8	10.1	1.21	0.84	0.20	0.85	8.6	1.0370	541†
	Cut No. 2	1385	1.80	86.6	9.9	1.23	0.95	0.13	1.19	8.76	1.0415	576†
	Cut No. 1	1420	15.80	86.7	9.7	1.29	0.95	0.03	1.33	8.94	1.0479	616†
Hexane-solubles		....	5.6	86.25	8.55	2.03	1.15	0.07	1.95	10.1	1.103	1630*; 850‡
CCl_4-solubles		....	9.4	84.4	7.6	2.24	1.33	0.97	3.46	11.1	1.116	2400†; 1660‡

* In benzene, at the boiling point.
† In benzene, at the freezing point.
‡ In naphthalene, at the freezing point.

TABLE 3

Waterman Analyses of Fractions of Dubbs Isopentane Solubles

Fraction	Refractive Index, $^{20}n_D$	Density, d^{20}_4	Specific Refraction, $^{20}r_D$	Molecular Wt.	Aniline Point, deg. Cent.	PERCENT BY WEIGHT		
						Aromatics	Naphthenes	Paraffins
10	1.549 (5)	0.9681	0.3289	270	41.0	36	16	48
9	1.573 (2)	0.9975	0.3303	345	42.1	45	8	47
8	1.58 (4)	1.0136	0.3318	344	40.5	47	1	52
7	1.58 (9)	1.0220	0.3297	350	32.5	51	2	47

tained at appreciably higher internal pressure of the solvent, aromatics being characterized by higher internal pressures than paraffins.[4] The determination of the average molecular weights for the maltenes presents no difficulties and they may be taken at their face value.

In Tables III and IV the Waterman[5] analyses are given for those cuts which were not too dark. Here again the greater aromaticity of the cracked fractions is apparent. It should be noted that owing to the dark color only a limited number of the cracked fractions could be examined. However in the maltenes of the 65 penetration cracked bitumen, the first fractions have already reached approximately the same aromaticity as the last fraction of the straight run.

It will be seen that the assumption based upon rheological characteristics and a high degree of dispersion, that cracked residues possess a more aromatic medium than straight run, is well supported by the above observations.

MOLECULAR WEIGHT DATA

The average molecular weights for cracked fractions given in Table I show the anomaly of becoming approximately constant while the carbon-hydrogen ratio and the density continue to increase.

It is difficult to say with what reliance the determined average molecular weights of such ill-defined mixtures as the isopentane-insoluble fractions can be accepted as absolute, for the solutions of these fractions in all probability depart appreciably from the ideal solution laws. However, they are considered to have a relative value, for a reproducibility within some 7 percent was readily obtained on any one sample, even after the lapse of several weeks. Also the determinations were made at several concentrations and the results rejected if the slope of the plot of apparent molecular weight against concentration was too great to allow reliable extrapolation to zero concentration. The values given in the tables are the extrapolated values.

Thus while the absolute values found for the final approximately constant molecular weights must be accepted with caution, the existence of a final constant value is in all probability real.

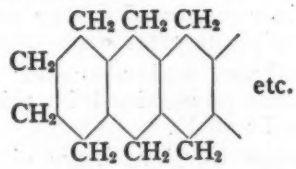
Rather surprisingly the reproducibility of the molecular weights even in different solvents of the cracked fractions was much superior to that of the corresponding straight material. Among the cracked products, values above about 900 were never obtained except with what were obviously unsuitable solvents, that is, solvents for which the slope of the plot of apparent molecular weight against concentration was too great or there was incomplete solution, gelation, etc.

With the straight-run samples, reproducibility on any given sample was good, but in the case of samples with a molecular weight above 1000, anomalies were observed. As in the case of similar determinations on coal hydrocarbons[6] the molecular weights seemed to vary from solvent to solvent, suggesting the possibility of some kind of polymeric aggregation due to a not very strong binding and readily reversible by a sufficiently powerful solvent.

The molecular weight data must be accepted therefore more as an interesting indication and as showing that the asphaltenes in low-level cracked residue for the particular Californian crude examined are of less molecular complexity than the uncracked ones.

THE CHEMICAL SIMILARITY OF CRACKED MALTENES, CRACKED AND UNCRACKED ASPHALTENES

Abraham makes the statement[7] that asphalts have a polycyclic character typified by an extended naphthene structure of the type



Mack[8] speaks of them as polycyclic compounds containing oxygen and sulfur, or both, in bridge or heterocyclic linkage.

Sachanen and Tilicheyev[9] remark that "Due to the fact that the presence of benzene derivatives with long side chains is improbable on account of their instability (to cracking temperatures), increased accumulation of naphthalene derivatives and other condensed aromatic hydrocarbons must be expected." They also point out that the extent to which condensed aromatic hydro-

TABLE 4

Waterman Analyses of Fractions of Straight Run Isopentane Soluble

Fraction	Refractive Index, $^{20}n_D$	Density, d^{20}_4	Specific Refraction, $^{20}r_D$	Molecular Wt.	Aniline Point, deg. Cent.	PERCENT BY WEIGHT		
						Aromatics	Naphthenes	Paraffins
19	1.5008	0.9075	0.3245	293	71.2	14	35	51
18	1.5136	0.9292	0.3238	373	77.6	18	26	55
17	1.5167	0.9343	0.3237	365	80.1	14	36	50
16	1.5170	0.9347	0.3237	391	77.8	18	32	50
15	1.5221	0.9452	0.3227	421	77.0	20	34	46
14	1.5231	0.9433	0.3239	413	77.8	20	30	50
13	1.5271	0.9493	0.3239	427	77.0	22	29	49
12	1.5301	0.9529	0.3242	438	75.4	24	28	48
11	1.5342	0.9593	0.3241	448	73.0	26	27	47
10	1.5362	0.9613	0.3245	448	72.2	27	24	49
9	1.5422	0.9690	0.3249	497	69.3	32	24	44
8	1.5481	0.9790	0.3245	489	64.9	34	25	41
7	1.5597	0.9934	0.3254	497	62.4	37	22	41
6	1.5693	1.0031	0.3268	520	59.3*	42	13	45
5	1.5783	1.0157	0.3269	539	56.6*	45	10	45
4	1.5862	1.0234	0.3280	524	55.3*	47	6	47
3	1.5946	1.0340†	0.3284	541	54.2*	48	5	47
2	1.6008*	1.0415	0.3288	576	53.4*	50	4	46
1	1.6060*	1.0479	0.3291	610	52.4*	53	1	46

* Extrapolated. † Interpolated.

[4] Hildebrand, "Solubility," Second Edition, Chapter 10, Reinhold Publishing Corp., New York City (1936).

[5] J. C. Vlugter, H. I. Waterman and H. A. von Westen, "Improved Methods of Examining Mineral Oils, Especially the High Boiling Components," *Journal*, Inst. Petroleum Technologists, Vol. 21, p 661 (1935).

[6] R. C. Smith and H. C. Howard, "Molecular Weights of Polymeric Substances in Catechol and Their Bearing on the Nature of Coal and Derived Products," *Journal*, Am. Chemical Soc. Vol. 58, p. 740 (1936)
R. C. Smith and H. C. Howard, "Equivalent and Molecular Weights of Humic Acids from a Bituminous Coal," *Journal*, Am. Chemical Soc., Vol. 57, p. 512 (1935).

[7] H. Abraham, "Asphalts and Allied Substances," Third Edition, p. 750, D. van Nostrand Co., New York City (1929).

[8] C. Mack, "Colloid Chemistry of Asphalts," Colloid Symposium Monograph, Vol. 10, p. 53, Chemical Catalog Co., New York City (1932).

[9] A. N. Sachanen and M. D. Tilicheyev, "The Chemistry and Technology of Cracking," p. 135, Chemical Catalog Co., New York City (1932).

carbons are formed is determined by the depth of cracking.

From the above and similar statements in the literature, it appears to be generally believed that asphaltenes, both cracked and uncracked, are highly aromatic bodies containing more or less condensed aromatic nuclei and that the degree of condensation increases upon cracking.

Now it is known that pure polycyclic compounds such as picene, crackene, and truxene are extremely insoluble in most solvents whereas asphaltenes readily dissolve in benzene, carbon bisulfide, etc. Their solubility in such solvents is not dependent on the presence of resins and the like, as they remain soluble after repeated solution and reprecipitation until the precipitating solvent remains colorless. It can therefore be concluded that in the asphaltenes, cracked or straight, the degree of condensation of the aromatic nuclei is unlikely to be very great although the number of rings present can be large.

In addition to the solubility in benzene, there is the observation that the cracked asphalts, usually assumed to be more highly condensed, possess rheological properties which can only be associated with a lyophilic colloid more highly dispersed than uncracked asphalt systems. These facts throw some doubt upon the conception of a condensed polycyclic structure for asphaltenes, particularly of the type postulated by Abraham.[7]

It has long been held that coal tars can be distinguished from petroleum tars by the fact that the latter resist attack by sulfuric acid. However, if asphaltenes are aromatics, it is surprising that they are unsulfonatable. The explanation appears to be quite simple. They are apparently so strongly protected by adsorbed oily material and resins that the acid cannot penetrate to the asphaltenes in a reasonable time. On the other hand, the highly purified asphaltenes prepared by the extraction method already described are readily sulfonated by fuming acid and rendered completely water-soluble.

Cracked maltenes are also sulfonatable with fuming sulfuric acid, but the straight-run material is not attacked. In fact, the original, untreated Dubbs residue used in the experiments could be sulfonated to almost complete solubility in water; only a trace of tarry matter remained undissolved. This indicates that the paraffinic groups shown by the Waterman analyses to be present are probably all attached to aromatic nuclei in the particular sample examined. In other words it appears that the maltene portion of the straight-run residues obtained from this crude oil contains wholly paraffinic molecules as well as paraffin chains attached to aromatic or naphthenic nuclei, whereas in the maltenes from the cracked residues no free paraffins exist, all paraffin groups being present as side chains of aromatic or naphthenic molecules. It would be interesting to investigate how far this is general and whether on cracking waxy crude oils the wax that is left may not be of a ceresinic aromatic character or the only truly paraffinic portions surviving.

Another extremely interesting observation is that all the different fractions from the particular cracked residue examined except the carbenes and carboids give, by sulfonation and liming, calcium salts which qualitatively are identical, having the same appearance, soapy qualities, and extreme solubility in water. Solutions of these salts all exhibit the same curious behavior on evaporation, first becoming syrupy and finally drying rapidly to a powdery consistency.

The above facts suggest that all the constituents of this cracked residue, except possibly the carbenes and carboids are closely related chemically and are built up of units of some simpler compound or compounds. Even the straight-run asphaltenes with average molecular weights of the order of 2400 in benzene may in the main be referable to such simpler units, and split down to them during sulfonation. It is difficult to conceive of a sulfonated molecule of around 2400 molecular weight giving the thin, clear, non-gel-like solutions in water that are obtained.

A TENTATIVE THEORY OF THE STRUCTURE OF THE CONSTITUENTS OF RESIDUES

The theory to be described is designed primarily as a contribution to the knowledge of the structure of asphalts and is presented not as a necessarily correct theory but only as one formulated during the normal process of development and subject to further investigation. Any theory of the structure of the constituents of bitumen must take into account what is known of the nature of their nearest relatives, the heaviest lubricating oils. The National Bureau of standards has concluded[10] that in passing from gasoline to oil the paraffin content decreases while the proportions of aromatics and naphthenes increase. The heaviest lubricating oils they examined were considered to comprise chiefly naphthenic molecules with one of more single, double, or triple, etc., naphthenic ring nuclei, with or without paraffin side chains, and aromatic molecules with one or more benzene, naphthalene, anthracene, etc., nuclei, also with or without paraffin side chains.

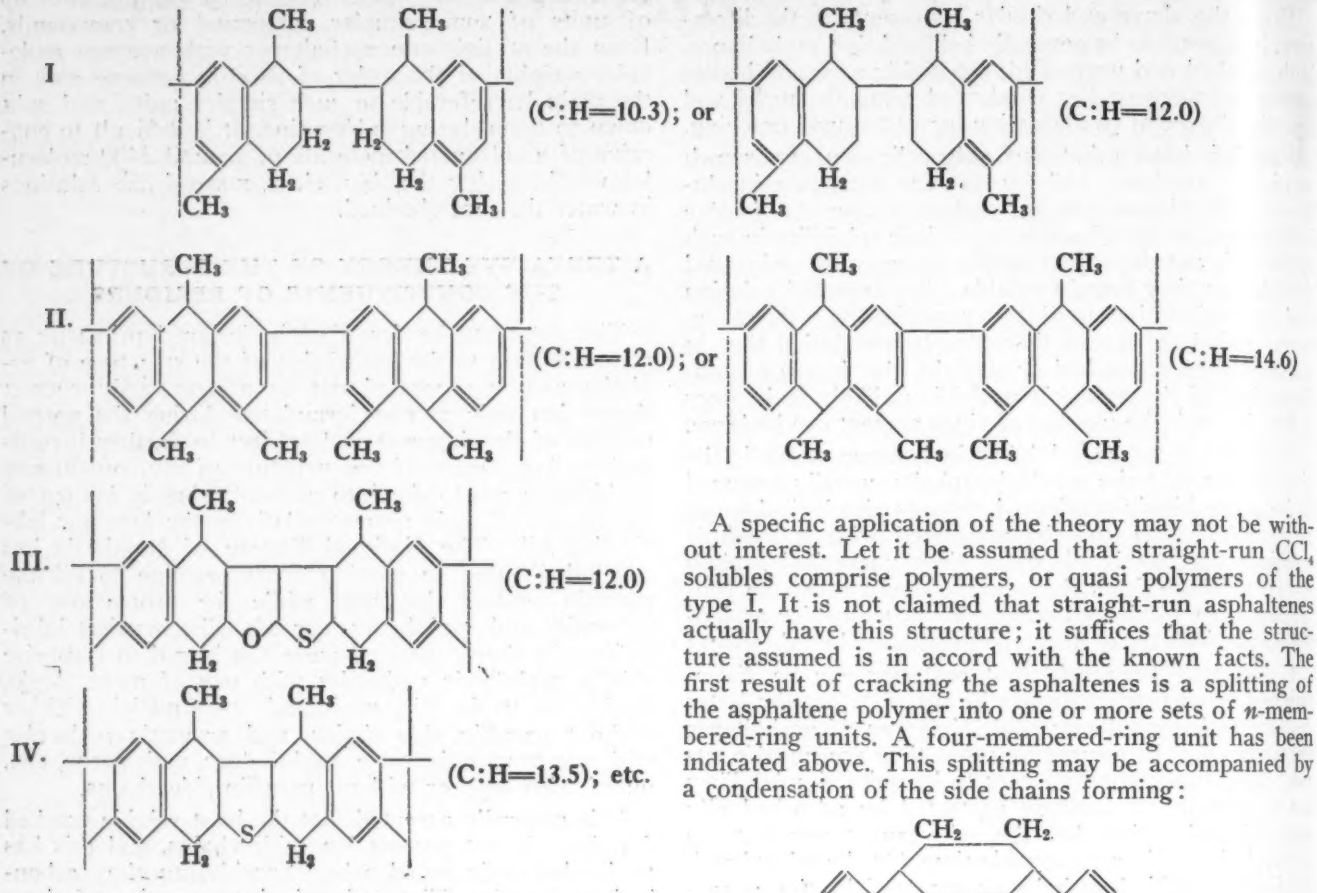
It is generally accepted that the least soluble cracked fractions do not possess long side chains, and this has to be borne in mind when considering the carbon-hydrogen ratios. The carbon-hydrogen ratios of these fractions are not high enough to permit the presence of large condensed nuclei corresponding to the molecular weights found. The alternative is units of relatively smaller nuclei strung together. Indeed, it is difficult to conceive of any other structure for the highly naphtho-aromatic asphaltenes of which the molecular weight is from 700 to 800, while the carbon-hydrogen ratio is only 13.5 instead of, for instance, 24.0 for decacyclene, of which the molecular weight is 450. This is particularly true for the straight-run asphaltenes, whose molecular weights are already about 2400 while the carbon-hydrogen ratio is only 11.1. Because of the similarity of the derivatives, such as bromides and sulfonic acids, of the various fractions, it is highly probable that the units which combine to form the larger molecules are limited to a few types that are very similar.

In the process of mild cracking such materials as resins or asphaltenes, the larger structures are broken down into simpler units. At the same time the remaining side chains may be lost, apparently as methane (for which there is experimental evidence), and sensible intradehydrogenation occurs with the possible liberation of gaseous hydrogen in small amount. As a consequence, some of the cracking products are not only less aliphatic (accounting for the rise in the carbon-hydrogen ratio) but also more condensed. Since the highly condensed aromatics with over four or five benzene rings are known to be only slightly soluble in even the best solvents, the decreased solubility of some fractions after cracking becomes understandable.

On the other hand the possibility, for the bulk of the products, of a simple breakdown very analogous to depolymerization accompanied by only slight condensation, is not excluded. The literature indicates that in general condensation requires more drastic cracking

[10] Rossini, "Fundamental Research on the Chemical Constitution of Lubricating Oil—A.P.I. Research Project 6," *Proceedings*, Am. Petroleum Inst., Vol. 16M, (III), p. 63 (1935).

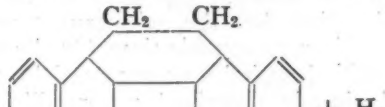
FIGURE 1
Structures of Resins and Asphaltenes



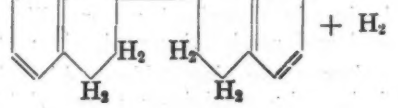
conditions than depolymerization, so that depolymerization could be the first step in the cracking of such materials as resin and asphaltenes. Under mild cracking conditions, therefore, as in the preparation of low-level residue from straight-run residue, depolymerization may be expected to be the preliminary reaction occurring among the bitumens accompanied or almost immediately followed by loss of side chains, and the constituents of such residues should be chemically similar and differ only as members in a homologous series differ in such properties as molecular weight, density, and length of side chains. In fact, the constituents of low-level residue up to and including the asphaltenes actually do behave in this way. The lower solubility of the cracked as compared with the uncracked asphaltenes can be accounted for sufficiently by the decrease in paraffinicity due to loss of side chains. The very small amounts of heavier fractions (carbenes and carboids) present in the low-level residue are to be ascribed to the effect of the condensation reaction which under low-level cracking conditions is apparently incompletely "screened out."

The matter given in the preceding pages, together with much data not conveniently presented here, appear to indicate that resins and asphaltenes may have structures such as shown in Figure 1. The above are more representative of cracked than straight-run asphaltenes in which there are probably longer side chains corresponding to lower carbon-hydrogen ratios. Although these structures have been represented as identical polymers, the possibility that the units of the resin and asphaltene molecules may be dissimilar is not excluded. Such molecules in which the units are unlike may be called quasi polymers.

A specific application of the theory may not be without interest. Let it be assumed that straight-run CCl_4 solubles comprise polymers, or quasi polymers of the type I. It is not claimed that straight-run asphaltenes actually have this structure; it suffices that the structure assumed is in accord with the known facts. The first result of cracking the asphaltenes is a splitting of the asphaltene polymer into one or more sets of *n*-membered-ring units. A four-membered-ring unit has been indicated above. This splitting may be accompanied by a condensation of the side chains forming:



At the same time the naphthene rings are dehydrogenated with an accompanying shift of hydrogen to the free bonds:



The final product is picene, molecular weight 278, only slightly soluble in benzene and somewhat more soluble in pyridine. It is improbable that as much hydrogen as is indicated actually escapes as gas.

Perhaps it is not without significance that picene has actually been isolated from a California cracked residue.[11]

While the above may appear to suggest a very simple and perhaps long-chain molecule, we do not pretend that such exists in asphalt. Rheological properties suggest a compact ball-like structure in which the above type of unit is probably rolled up upon itself or so connected that some of the units are close to each other in spatial configuration and are more or less united by the residual valences and polar forces in the unsymmetrically substituted rings.

ACKNOWLEDGMENT

The acknowledgments of the authors are due to the Directors of the Shell Development Company for permission to publish the above work carried out at their laboratories in Emeryville, Calif.

[11] C. Graebe and J. Walter, "On Picene," *Berichte*, Vol 14, p. 175 (1881).

The Relationship Between Laboratory Column Distillations and A. S. T. M. End Points

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Petroleum Engineering School, University of Oklahoma

THERE has been a gradual improvement in the design of laboratory distillation equipment for the analysis of crude oil and various petroleum fractions in the last two decades. The greater part of this advance has taken place, however, during the past 11 years. Peters and Baker[2] were among the first to design a column for accurate high-temperature distillations. The type of column designed by these investigators was modified in such a way that heavier petroleum fractions could be distilled under vacuum in order to prevent cracking from taking place. Since 1926 the old type Hempel columns have been abandoned to a large extent in favor of the taller true-boiling-point columns most of which range from 4 to 8 feet in height. Along with the development of the high-temperature column came the design of a low-temperature column by Leslie and Podbielniak for the analysis of natural gas and natural gasoline fractions.

Although these taller columns gave more accurate analyses, their results must be correlated with the standard A.S.T.M. distillation test which is used as the basis for most control work. A. J. Good and A. J. Connell[1] have made a study of the relationship between the A.S.T.M. end points and true-boiling-point-cut temperatures on a large number of oils distilled in various laboratory columns. These investigators came to the conclusion that A.S.T.M. analyses cannot be used as a criterion of effectiveness of separation without consideration of the temperatures involved. In view of the scarcity of published data on this subject, further work has been undertaken in the Petroleum Engineering Laboratories at the University of Oklahoma in order to compare the behavior of two different types of laboratory distillation columns against the A.S.T.M. tests.

EXPERIMENTAL APPARATUS

In Figure 1 the high-temperature column made of Pyrex glass is shown on the left hand side of the photograph. This column is similar in design to the one used by Peters and Leslie[3], and is equipped with a 1-liter flask at the base. The column measures 1¼-inch I.D. and 5 feet in height. It is packed with thin-walled glass nipples averaging about ¼-inch in diameter and 1/3-inch in length. The column is wrapped with a nichrome wire wound spirally around the full length of the column. The air jacket is provided with openings so that compressed air can be blown through the annular space in order to increase the amount of reflux, if necessary.

In Figure 2 an iron still is shown, equipped with an unheated fractionating column which is about 6 feet in height. In carrying out the crude oil distillations in this still it was found that the cooling coil could not be used at the top of the column due to the absence of any heating element around the lower part of the column. In making distillations with this still, reflux had to be provided from the

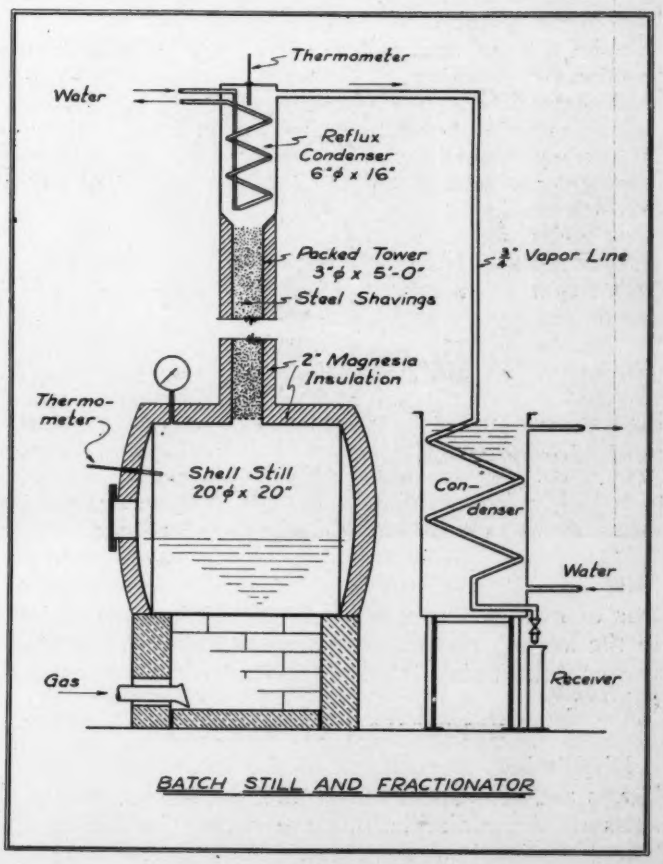

FIGURE 2
Iron Still with unheated fractionating column

Present addresses
* Gulf Oil Corporation, Port Arthur, Texas.
† The Texas Company, Port Arthur, Texas.
‡ Phillips Petroleum Company, Bartlesville, Oklahoma.

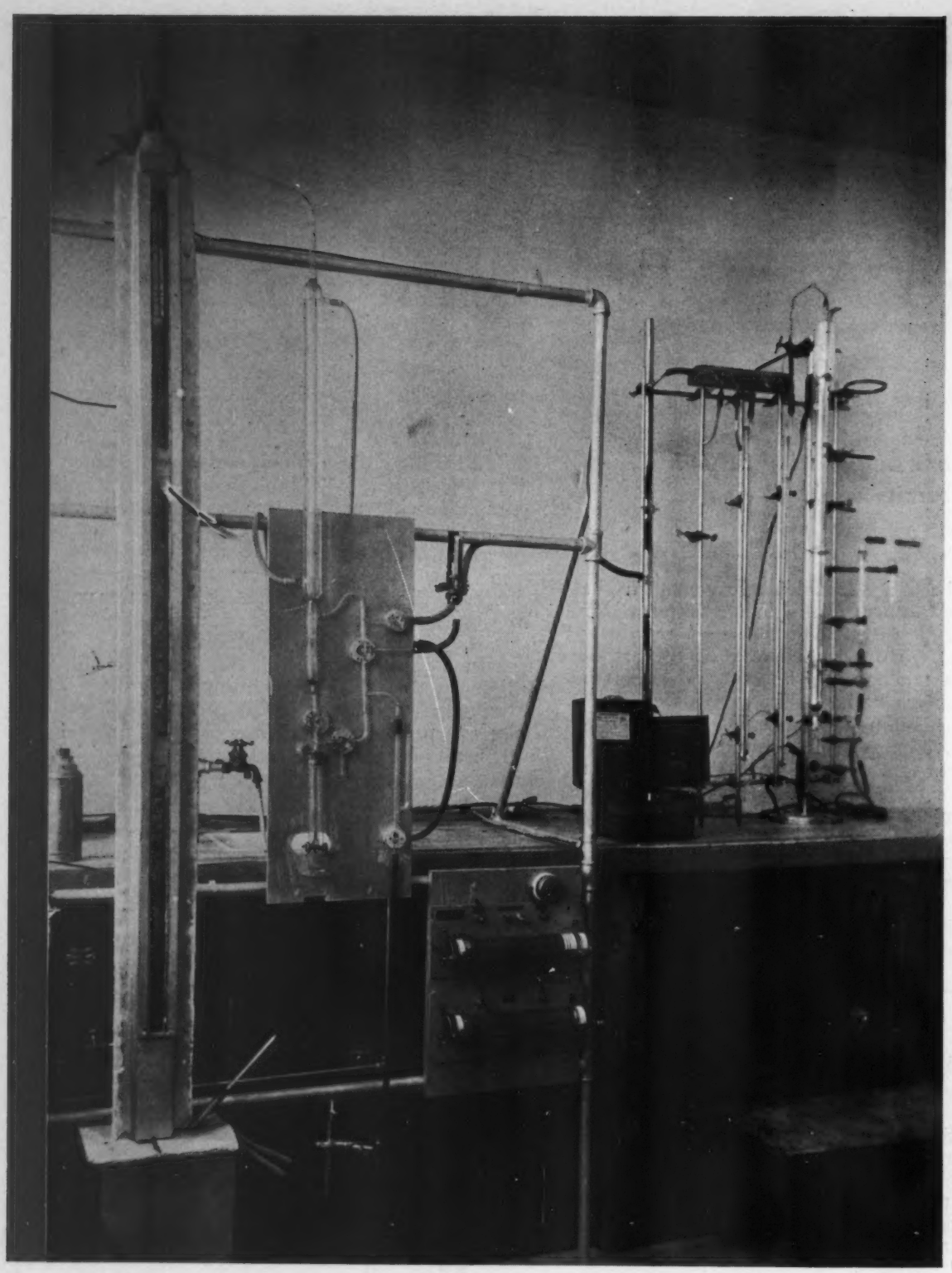

FIGURE 1

Left to right: High temperature true boiling point column and Podbielniak low temperature column.

loss of heat due to radiation through the insulation in the lower part of the column and the loss of heat through the uninsulated section near the top of the column.

DISCUSSION OF RESULTS

Distillations were made in the iron still on East Texas and Tonkawa, Oklahoma, crude oils. The results of these distillations are shown in Figure 3. These distillations were carried out at the rate of about 100 cc. per minute. At this rate the radiation losses would give a reflux ratio of about 1.6 of reflux to 1.0 of product when the cut temperatures at the top of the column were running around 200°F. As the distillations proceeded to higher temperatures, the reflux ratio naturally increased until at 450° F. cut temperatures, the calculated reflux ratios amounted to about 3.2. The A.S.T.M. distillations on the several gasoline fractions taken from the distillation of the East Texas and Tonkawa crudes are shown in Figures 5, 6 and 7, respectively. In Figure 4 the relationship between the true-boiling-point-cut temperatures and the A.S.T.M. end points on these two crude oils is shown.

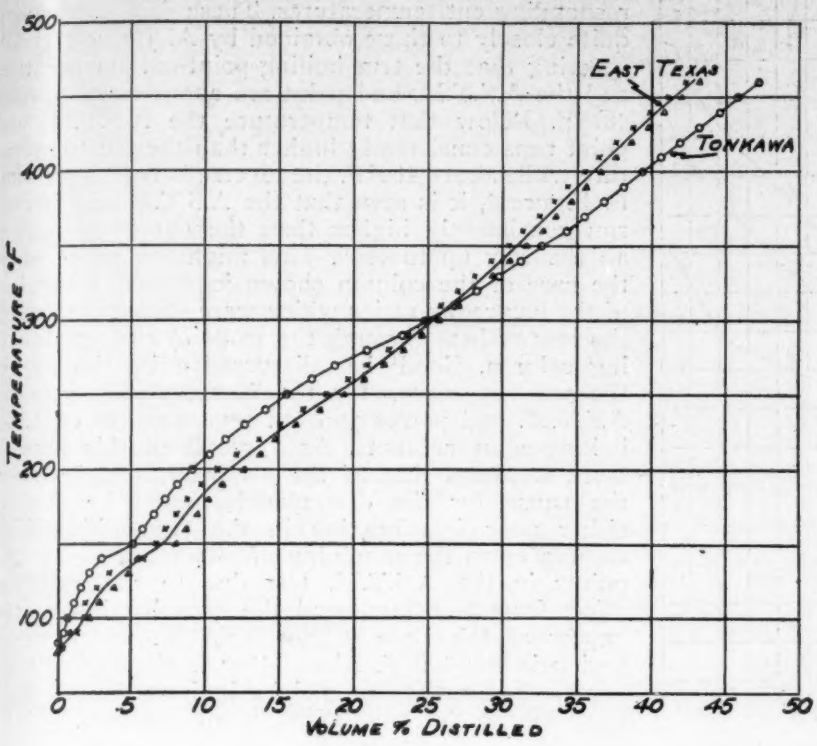

FIGURE 3

Crude Oil Distillations made in shell still shown in Figure 2

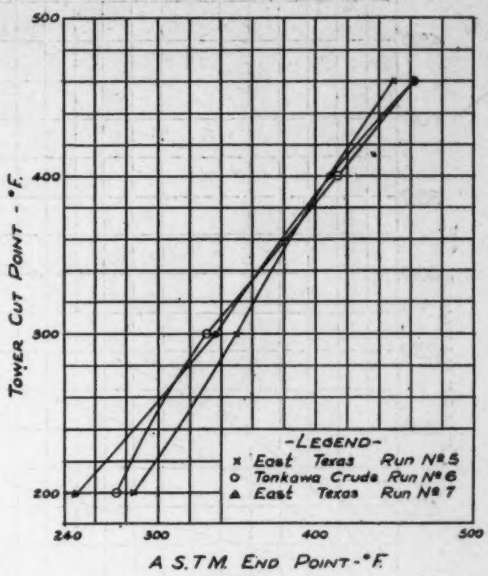

FIGURE 4

Relationship T.B.P. Cut temperatures to A.S.T.M. End Points. Degrees Fahrenheit.

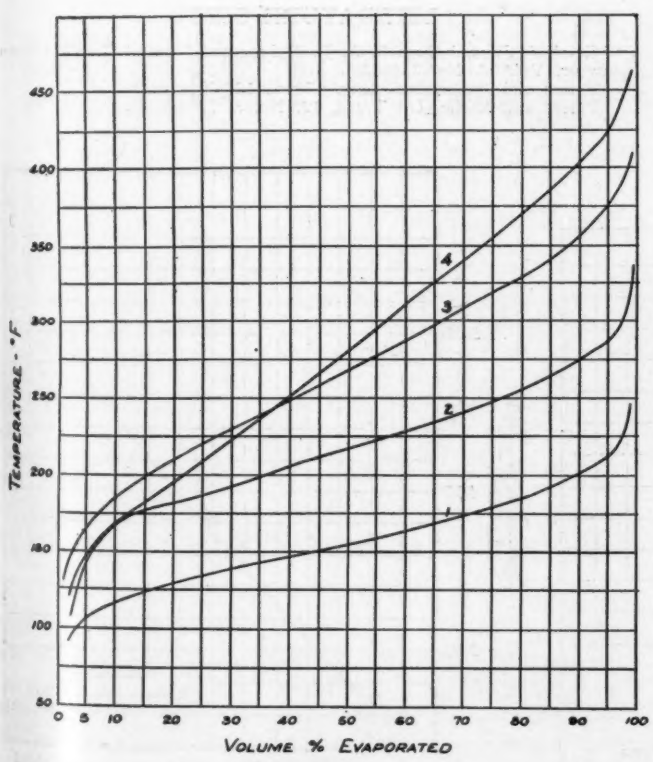

FIGURE 5

A.S.T.M. Distillations of Shell Still tower cuts, Run Number 6, Tonkawa crude. (1) 200°F. cut. (2) 300°F. cut. (3) 400°F. cut. (4) 450°F. cut.

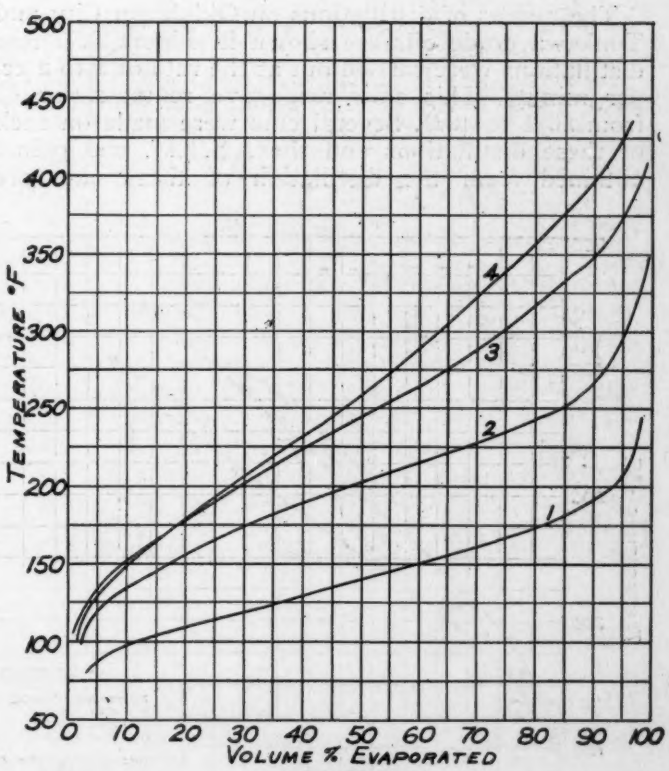

FIGURE 6

A.S.T.M. Distillations—Shell Still Tower Cuts Run No. 5 East Texas Crude. (1) 200°F. cut. (2) 300°F. cut. (3) 400°F. cut. (4) 450°F. cut.

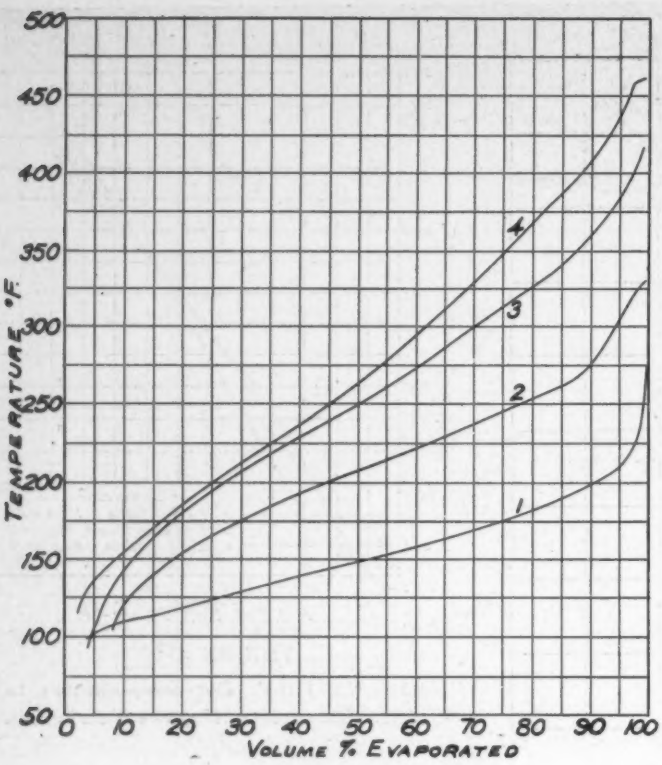

FIGURE 7

A.S.T.M. Distillations—Shell Still Tower Cuts Run No. 7 East Texas Crude. (1) 200° F. cut. (2) 300° F. cut. (3) 400° F. cut. (4) 450° F. cut.

DISTILLATIONS IN LABORATORY GLASS COLUMN

The results of distillations on Oklahoma City and Tonkawa crude oils are shown in Figure 8. These distillations were carried out at the rate of 1 to 2 cc. per minute. This slow rate gave reflux ratios of from 20/1 to 40/1. Several cuts were made on each of these distillations and the A.S.T.M. end points obtained from the distillation of these cuts are shown in Figure 9, in their relationship to the corresponding cut temperatures. These data correspond quite closely to those obtained by A. J. Good et al[1], showing that the true-boiling-point-cut temperature and the A.S.T.M. end point are about equal around 400°F. Below that temperature the A.S.T.M. end point runs consistently higher than the cut temperature while above 400°F. the reverse is true. Referring to Figure 4, it is seen that the A.S.T.M. end points run consistently higher than the cut temperatures all the way up to 460°. This might be expected in the case of the column shown in Figure 2 in view of the low reflux ratios which were obtained through the loss of heat through the walls of the fractionating column. Good[1] has discussed quite thoroughly the various causes for the discrepancies between A.S.T.M. end points and cut temperatures on true-boiling-point columns. As a result of this present work it seems that at the lower-cut temperatures the cause for the discrepancies must be due to either poor fractionation in the true-boiling-point column or to the obtaining of abnormally high end points in the A.S.T.M. test due to the radiation effect from the flame and the superheating of the vapors at the close of the A.S.T.M. test, for the region below 400°F. Above 400°F., the higher temperatures for the cut points indicate better fractionation in the true-boiling-point columns.

ACKNOWLEDGMENTS

The authors wish to express their appreciation to Messrs. Joe A. Caviezel and Carleton Shead who assisted in obtaining the distillation data; and to Dr. R. L. Huntington for his help in directing the work.

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[2] Peters and Baker, Ind. Eng. Chem., 18, 69 (1926).
[3] Peters and Leslie, Oil Trade, 17, No. 3, 37 (1926).

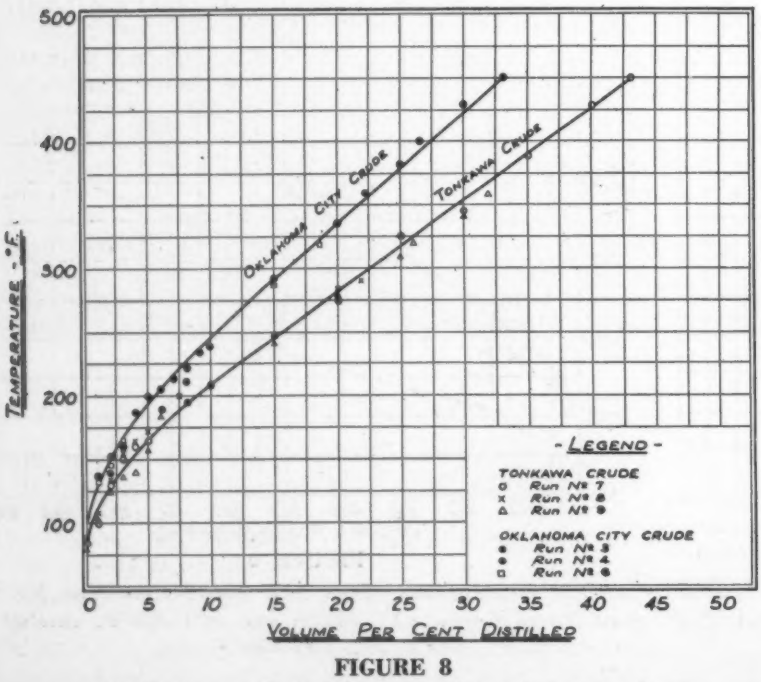


FIGURE 8

Distillations made in laboratory glass columns as shown in Figure 1.

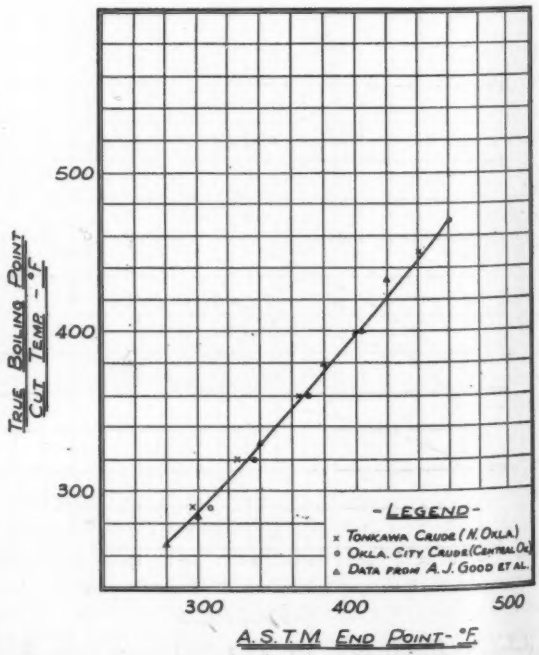
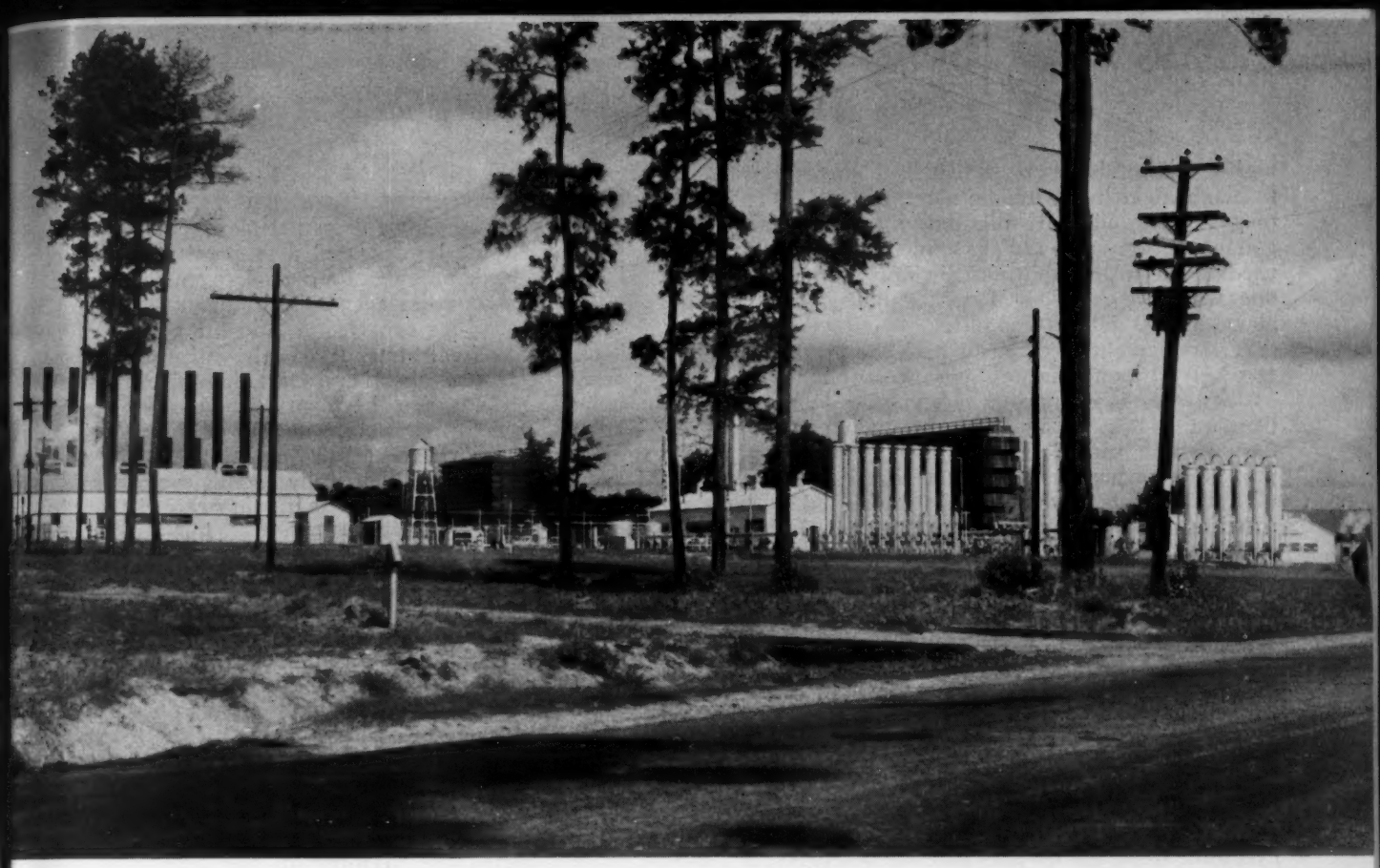

TRUE BOILING POINT CUT TEMP - °F
A.S.T.M. END POINT - °F
- LEGEND -
TONKAWA CRUDE (N. OKLA.)
OKLA. CITY CRUDE (CENTRAL OK.)
DATA FROM A. J. GOOD ET AL.

FIGURE 9

Relationship T. B. P. cut temperatures to A. S. T. M. end points °F. Tonkawa and Oklahoma City crudes distilled in Pyrex Column (See Figure 1).



United's Myrtis Gasoline Plant Enlarged Three Times

SINCE it has been necessary to increase the size of its gasoline plant at Myrtis, Louisiana, twice since it was originally built in 1933, United Gas Public Service Company is operating the largest natural gasoline plant in Louisiana. The first section of this plant was built to process dry gas from Rodessa when that area was strictly a gas field. Eight absorbers were installed to handle the gas at that time, each measuring 3 feet in diameter and 30 feet in length, with a combined capacity of approximately 40,000,000 cubic feet daily. This unit is a complete plant in itself, having the usual type of distillation apparatus, boilers and accessories and a finished product capacity of about 30,000 gallons of natural per day.

In 1935, increased gas production and demand for industrial and domestic fuel made it necessary for the second unit to be built, since the first unit was designed for a definite load and operating characteristics, and had been operating at capacity since its construction. The second plant contains 3 absorbers, measuring 4 feet in diameter and 30 feet in length, with a combined capacity of about 45,000,000 cubic feet of gas daily. This unit, like the first installation, is a complete plant within itself, operating independently of the first unit, but discharging its residue gas into a common distribution system. The combined load of both units after the second plant was built was about 95,000,000 cubic feet, and a natural gasoline output of approximately 85,000 gallons daily.

During the summer of 1936, after the present Rodessa oil field had been extended to cover a large acreage, gas production leaped to such a volume that it became necessary for the company to enlarge the gasoline plant again. Many wells drilled in Louisiana and the Texas side of the field provided sufficient gas to justify the erection of a modern compressor station to handle the gas from the traps and separators. The flowing pressures, combined with production practice, did not warrant handling it direct from the field to the gasoline plant. Approximately 200 wells have been drilled in the oil producing section of the Rodessa field since oil was discovered making available about 60,000,000 cubic feet more of gas daily to be handled by the Myrtis gasoline plant.

All gas passed through the plant and each of the three units comes into the absorbers at sufficient pressure that it can be transported from the plant without being compressed again. Well pressures are sufficient to provide this head on all gas produced from strictly gas wells, and shut-in pressures, permitting the proper choke for allowable production on the oil wells in the field would permit this also, but stabilization to a certain extent is desired of the oil, hence the need for the compressor station.

The equipment built for the third plant includes 3

high-pressure absorbers, 4 feet in diameter and 30 feet in height, which can handle about 55,000,000 cubic feet of gas daily, and the distillation unit can produce 55,000 gallons per day of raw gasoline. Two stabilizers are used in this plant, splitting the raw production so that one will receive about 40,000 gallons, and the other about 85,000 gallons per day.

A large quantity of drips and compression gasoline is received from the new compressor station and its system of lines, most of which is off color, particularly the liquids brought in from the main gathering lines. Compression production at the compressor station proper is normally clear enough that it can be included with the plant production and stabilized without rerunning. Due to contamination with crude which is pulled over from the oil and gas separators, some crude is included with the main line drips, which cause the gasoline to have such a high end point that rerunning would be necessary, if the color was good.

When the volume of gas exceeds demand the present capacity of the Myrtis plant, part of it may be by-passed around the absorbers by opening semi-automatic lubricated plug valves. The controls on this system are hydraulic and can be manipulated from the plant yard, even though they were installed at some distance from the absorbers. A control panel at a convenient point in the plant permits, by turning the three-way cocks on the hydraulic lines, the by-pass gates to be opened or closed as desired without making a special trip to the manifold station.

All of the absorbers are equipped with automatic liquid-level controllers, maintaining a sufficient amount of absorption oil so that a liquid seal will always be maintained in the base of the columns. Most of the controls for this purpose are of the "kidney valve" type, directly connected to valves in the oil outlet lines connected to the mineral seal header. Oil is circulated by motor-driven centrifugal pumps, steam turbine-driven and gas engine-driven triplex piston pumps. All of which must be operated at a pressure sufficient to handle a back pressure of over 450 pounds, gauge.

Vapors from the accumulator and stock tanks are recompressed for processing, and the gas is delivered back into the main lines to mingle with the residue gas from the regular absorbers. Oil field and return tubular boilers are in use in the plant, assembled in two boiler houses, which furnish steam for processing the rich absorption oil drips and to operate the steam pumps. Water is produced plentifully from deep wells in this part of Louisiana which has a comparatively low hardness.

Water cooling is obtained by the use of two louvre towers, one recently installed being of the newer type which does not lose water from wind velocity. However, windage is not experienced to any great extent in this part of the country, and cooling depends largely upon the size and location of the cooling equipment. Condensing and oil-cooling sections, of the atmospheric type, are installed in the lower part of the towers, with water circulated from the louvre tower basin over the top in the usual manner. Exceptionally low temperatures are said to be obtained with the new tower for this part of Louisiana, approximating wet bulb conditions during the majority of the time.

Loading of the finished products is carried on as needed instead of storing large quantities of gasoline, using a 20-car rack on a spur not far from the plant. The rack was built with the idea in mind to facilitate loading of any number of commercial grades of gasoline at one time. To accomplish this result, several lines were laid to the rack and connected to individual headers on each side of the structure.

Birdseye view of part of the Myrtis, Louisiana, gasoline plant of United Gas Public Service Company.

BECAUSE they require little attention, regulators are frequently neglected. When given the care to which they are entitled, satisfactory service results.

Maintenance of Gas Regulators

E. C. McANINCH
Oklahoma Natural Gas Company

LONG DISTANCE transmission of gas with its necessarily high pipe line pressure, together with the necessity for reducing this pressure to a point where it can be safely and economically distributed as well as properly measured, has placed upon gas regulators a responsibility that is of major consequence. Therefore, their maintenance is of first degree importance.

Fortunately a regulator is a device that does not require an exceptional amount of attention. This very fact, however, causes them to be neglected and ignored ofttimes until their condition is such that pressure regulation has completely ceased to be.

The safety factors that enter into the distribution of gas in a community demand a guarantee that the pressure on gas lines in that area shall never become excessive. Gas measuring instruments function properly only when pressure conditions are as they should be. Many other circumstances enter into a demand for perfect pressure control and regulation.

As a result, the behavior of its regulators is of utmost concern to every gas company.

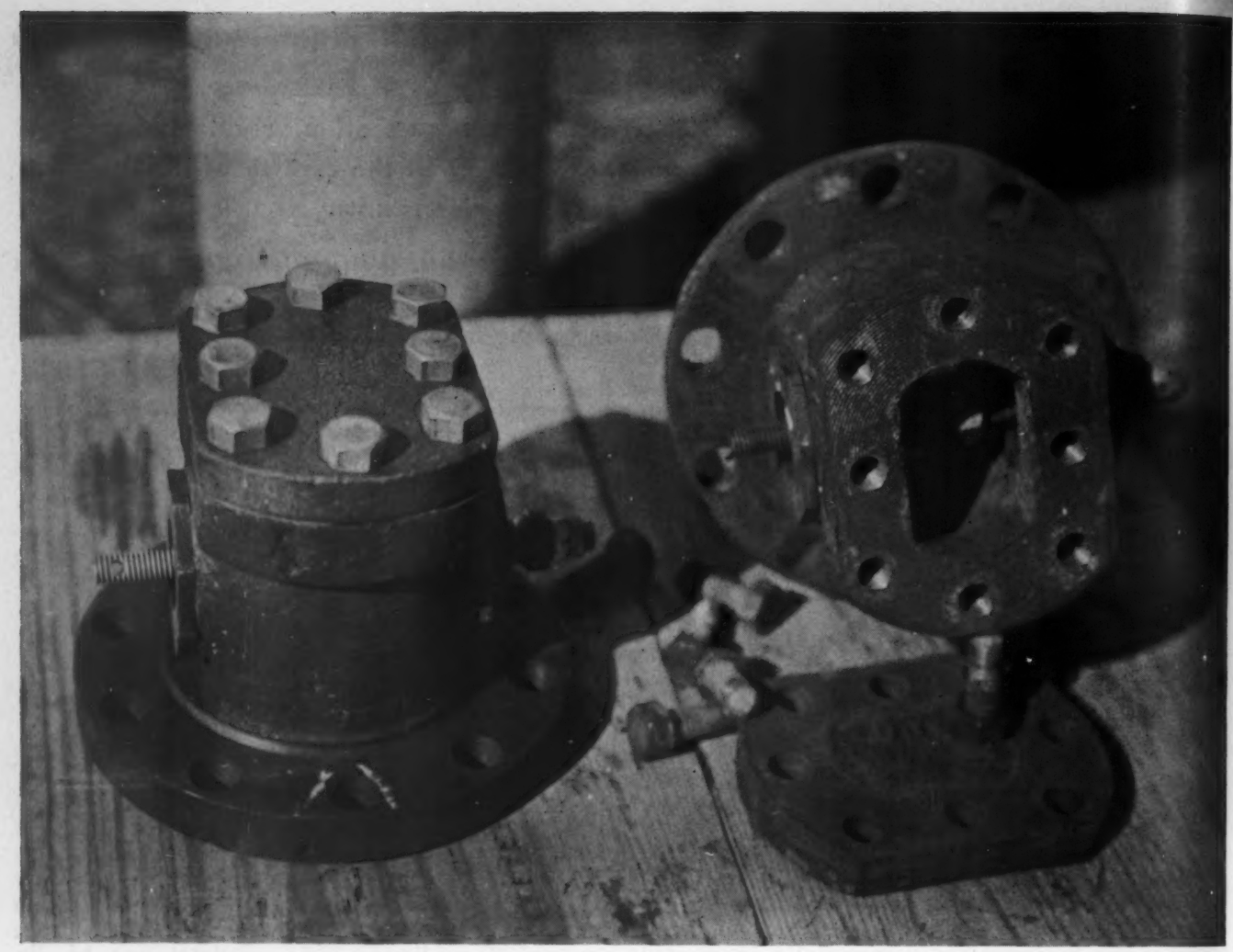
Regulators, just as all other instruments of gas control and measurement, should receive periodic attention. No set rule can be laid down as to how often this should be since conditions of operation are so greatly different. A careful study of the conditions under which a regulator is operating will show the period of time for which, ordinarily, it can be expected to function properly. A person can say safely, however, that no regulator should remain in service more than a year without being submitted to a thorough inspection and cleaning, with the replacement of such parts as is necessary.

When discussing the maintenance of regulators, it should be understood that installations are made properly and that the regulator itself is the proper size. Unless these conditions exist, proper regulation cannot be depended on, no matter how excellent the mechanical condition of the regulator may be. By proper size, we mean that the regulators should be large enough to meet the maximum demands made upon them, and yet small enough to control the minimum load. When this cannot be done through the use of a single regulator, a dual setting should be made.

Assuming these conditions, regulator troubles confronting the man responsible for their maintenance usually find their source in improper fitting valves and seats, binding of some of the working parts, wrong size of head used, or in loose connections and working parts. These are mechanical problems and to them should be added the ones of pulsation or pumping and freezing.

VALVE AND SEAT FITTINGS

In any line where gas is transmitted there is a certain amount of foreign substances, such as water, oil, gasoline, rust, dirt and pipe scale. These substances are carried through the lines until some obstruction is met. Here they lodge until the force of the traveling gas becomes great enough to dislodge them. A regulator is an ideal example of such an obstruction. When the gas is comparatively free of dirt and moisture its effect upon a regulator is small, but when a great deal of dirt and moisture is present, it is either lodged against the valves and seats or forced on through the regulator. When anything lodges on either the valves or their seats, the regulator cannot shut off the flow of gas, and pressure will build up to a higher point than is desired. The same result is had when gas forces these particles through the regulator and causes the valves and



The broken stem on this regulator inlet bowl is of minor expense itself, but may occasion a shut down, and thereby become a liability. The stem here was drilled for a pin. A drop of oil now and then, or more care in adjusting could have saved shut down and replacement cost.

seats to become rough and pitted. When this happens it is necessary to re-seat the valves. Study of the regulated pressure charts will show when this condition exists. If, during normal periods of operation, the pressure holds constant and then starts to build up when the load decreases or is shut off, a person can be sure that for some reason the valves are not completely closing. When grinding valves, it is necessary that both the valves be fitted at the same time. This may seem an unnecessary precaution, yet attempted fitting of first one valve and then the other has been known to occur.

In passing judgment upon the ability of a regulator to completely shut off, it should be remembered that if hard seats are used, it is practically impossible to shut off all flow of gas. Where this is demanded of a regulator, soft seats must be used.

WORKING PARTS MAY BIND

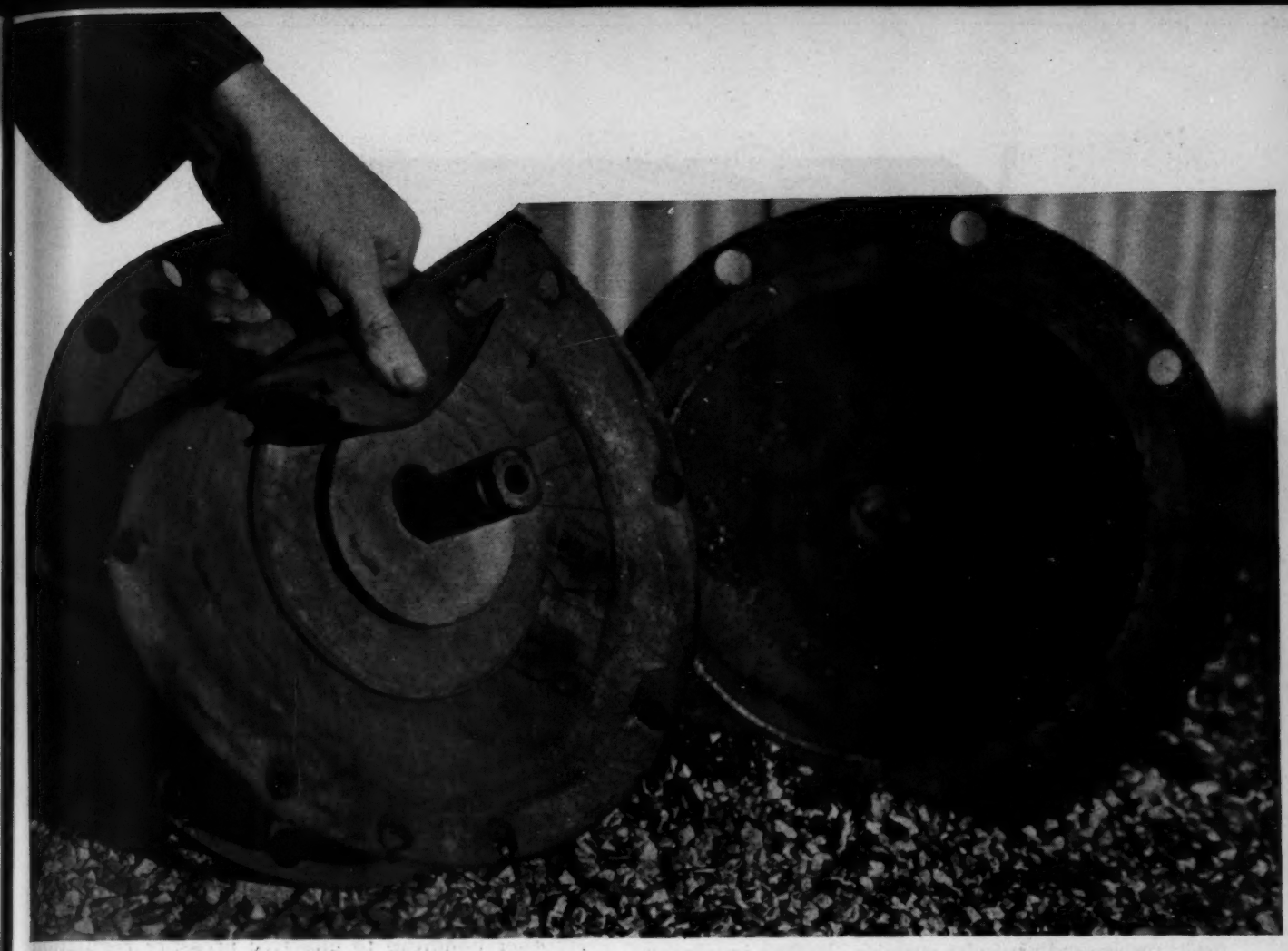
The second named cause of imperfect pressure regulation manifests itself in a different manner. Binding in any of the working parts of a regulator causes it to be insensitive to small pressure changes on the discharge side. Consequently, when the discharge rate is increased rapidly the pressure will drop several pounds before the enlarged friction of the bind is overcome and the valves open. They then open suddenly and gas passes through the regulator more rapidly than it would if the regulator were working properly. The pressure builds up too fast and the valves close. The result is a very uneven pressure.

The cause of binds is a misalignment of working parts. Either the valve guides are not true or they are corroded and dirty, or the valve stem is out of line or dirty and binding in its stuffing box. Paint on either the valve stem or the lever pin will have the same effect as a bind. Still another cause of sluggish regulators is an obstruction of the breather hole in the diaphragm case. This breather hole must be kept clean.

When a sticking or sluggish condition exists it is advisable to make a close inspection that the valve stem is straight and in perfect alignment with head and valve connections. If the valve stem has been bent and is binding, it should be replaced. Be sure that there is no paint on the stem or other working part. Repack the stuffing box with properly lubricated packing and do not tighten any more than is absolutely necessary.

INSTALL PROPER SIZE HEAD

The size of the head on a regulator is very important. Within limits, the larger the head the more sensitive the regulator to changes in the controlled



Diaphragms may become brittle through drying, and may crack; or may become deteriorated through contact with moisture. Operating fatigue can only make replacement necessary after long service, but it is necessary to inspect regulators frequently to assure correct pressures and continuous operation.

pressure. The smaller the head the greater the variation in discharge pressure which may be expected. Increased sensitivity with the use of a large head will not be obtained when, in order to maintain the desired outlet pressure, an excessive weight must be placed on the weight lever. This is an unwise practice because in addition to decreasing the sensitivity of the regulator, the lever pin may be sheared, the diaphragm bursted or the lever bent. When any of these things happen the regulator will not operate at all. In determining the size head to be used, it is advisable to follow the recommendations of the manufacturer.

The size of the head determines the diaphragm material which should be used. With high pressures and a small head, a heavier, less flexible diaphragm is used, and when the desired pressure is lower and a larger head is used, a more flexible material is needed. Use of a light material where high pressures are needed is dangerous because of its inability to withstand them. Heavy material used when lower pressures are desired lessens the responsiveness of the regulator to pressure changes and nullifies the use of the larger head.

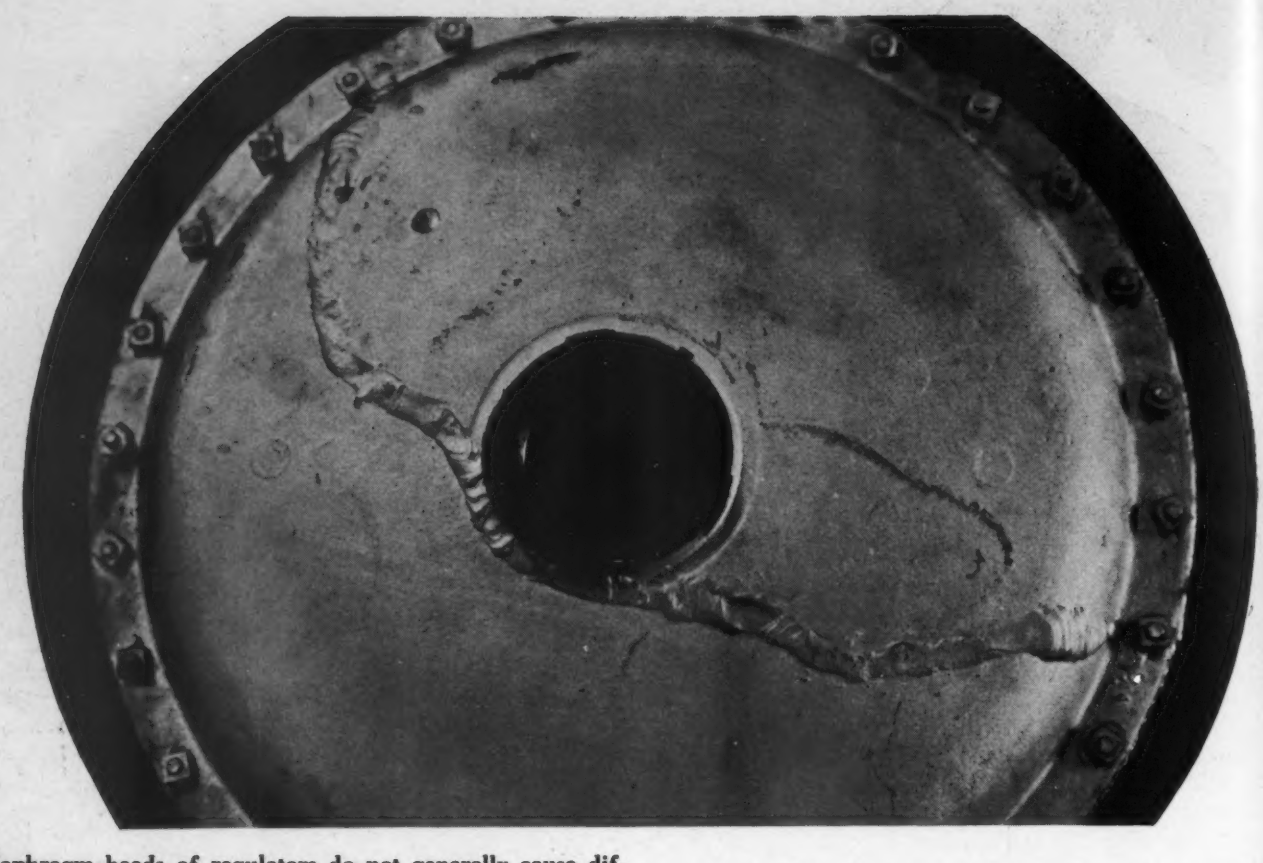
CORRECT INSTALLATION NECESSARY

Correct installation of a diaphragm is necessary if a regulator is to work properly. If the diaphragm is too tight, pressure exerted upon it will not completely close the valves and will cause a result similar to improperly seating valves. When re-diaphragming a regulator, be sure that the valves and the weight lever are in a wide open position at the time the diaphragm is punched and bolted in the case.

Loose connections cause both uneven regulation and chattering in the regulator. A very little play in the connections between the valves and the stem causes a fluctuation in the outlet pressure. This is due to a "lost motion" travel in the weight lever before the valves lift from their closed position. This play also will permit a vibration of the valves and the valve will chatter up and down. Sometimes the valve guides fit loosely and allow a vibration of the valve that causes it to chatter. The first cause may be remedied by inserting a thin tin washer between the loose connections. The second may be stopped by spreading the valve guides slightly so that no lost motion there is permitted. This may be done by tapping the bottoms of the guides with a hammer. If the valve guides fit very loosely it is best to install new valves.

STOP REGULATOR PUMPING

Under certain conditions of operation, regulators will "pump." This action is so called because of the regular up and down motion of the weight lever when it exists. This condition is most often found when successive pressure cuts are made with the regulator settings close together. Single regulators have been known to pump, however. Turbulent flow conditions are the cause of pumping. This turbulence causes an uneven pressure to be exerted upon the diaphragm, causing the valves to intermittently open and close. Several solutions for this problem have been suggested, and the use of any of them is a



Diaphragm heads of regulators do not generally cause difficulty, or demand particular attention. In their installation, due to the number and spacing of bolts around the circumference, it is necessary to tighten the head evenly, otherwise the part may be broken.

matter of personal preference and successful application. Sometimes the pumping is eliminated by reducing the size of the restriction in the diaphragm piping. This lessens the responsiveness of the regulator to sudden pressure changes. Pumping is sometimes stopped by connecting the diaphragm piping a greater distance from the regulator. Changing the size of the head is sometimes a remedy. Still another method is the installation of a control disc in the line just beyond the diaphragm piping connections. This allows the gas to "pack up" and thus prevents surging tendencies. This control disc should be sufficiently large, however, that an excessive pressure drop across it does not occur at high rates of flow.

Another handicap to effective pressure regulation is that of freezing. As gas passes through a regulator it is cooled. When this cooling process reaches the point where liquids are condensed out of the gas, freezing occurs. This happens most often when a great difference exists in the inlet and outlet pressure. Ordinarily it is prevented by heating the gas before it goes into the regulator, so that the resulting expansion does not cool the gas to the point where freezing occurs. Freezing in the regulator causes a binding of the working parts, and the substitution of center valve guides will aid materially. Freezing may occur in the diaphragm piping, especially if there are any leaks in either the connections or the head. To prevent this it is essential that no leaks exist.

When regulators are inspected periodically and given the attention to which they are entitled, then they will give the satisfactory service and regulation that is claimed for them.

Taking Stock

L. C. TRESCOTT

PART 2

IN the space of this article it is not possible to cover all of the small points that may cut down efficiency and break down organization. But if a job is carefully and conscientiously carried out, it will dig for the details peculiar to the operation it covers. The important thing is that it is to be a taking of stock, a determination of conditions whether they be good or bad. And from the pictures thus laid out, steps may be taken to put the plant and the organization on a better working basis.

IN a shop manufacturing motor cars, radios, or collar buttons, job analysis is a relatively simple matter. A man is usually limited to one series of production operations conducted on one machine. He is not called on to consider either the work that has been done on the material before it reached him or where it will go and how it will be treated when it leaves him. Generally he has no responsibility for the upkeep of his machine, and there are no hazards involving a particular responsibility for the safety of himself or his fellow workers. His job is generally to follow out a fairly clear-cut set of routine operations skillfully and according to set standards of quality and quantity. He is seldom called on to do any real planning or to use any considerable judgment in connection with his job.

In the refinery there are few jobs that fall within as closely prescribed limits as the above. To carry out refinery work requires that some judgment be applied to each job as it comes along. In order to fit, a man must have some overlapping knowledge of what has happened to a stock before it reaches him and what will be done with it after it passes on. Even though this movement of stocks be carefully provided for, still the men immediately concerned with it have responsibilities to others outside of their own departments, or their own immediate operations. More often than not the men on the job do the planning and the handling as a matter of course. Here the operator uses his own best judgment, and if something goes wrong he is held directly responsible.

While refinery operations are far from carrying the hazards of explosive plants or of some chemical plants, the operation of stills, cracking operations, etc., all offer opportunities for explosions and fires which may cause loss of life or considerable property damage. It is the responsibility of the man on these jobs to see that they are so operated that flashes do not occur or if they do happen that they are handled so that little damage is done.

Another essential item which must be considered in connection with a job analysis or a job survey is the actual physical work demanded by the job and the conditions under which this work is performed.

In classifying a job there are three main points to be considered.

1. The responsibility attached to the job.
2. The training required.
3. The physical work required.

Responsibility may take several forms. An operator may be held responsible for the operation of expensive equipment, as in the case of stillmen or operators in charge of large stills, the newer treating plants or dewaxing plants. Or a man may be responsible for the movement of large and varied quantities of oil as in the case of a pumper in charge of a large main pump house. There is a tendency to regard the first responsibility higher than the second and to rate the jobs accordingly. It must be borne in mind, however, that the operation of stills and other modern equipment is largely automatic. The principal requirement in handling this responsibility is a reasonable vigilance. More shifts than not pass with no changes in operation. There are few times when emergencies must be met. It is true that the operator must have some training and experience, but at the same time his instruments warn him of changes in the running and come pretty near telling him what to do. His records are seldom complicated and as a rule his operations call for but a small amount of physical work, especially work of a type which takes him out in all kinds of weather.

On the other hand, the man in the pump house has equipment under his care whose value is only a small fraction of the value of the still, the treating, or the dewaxing plants. The chances of any considerable damage to it are small. In the course of a year, however, quantities of stocks far higher in value than any plant equipment pass through his hands. He is called on to plan and put into effect the handling of these stocks. No indicators guide his work. He does not even have a map of the lines he uses. He must not only know the niche he occupies in his own department but he must have a fair idea of the stocks, the lines and the equipment of those

with whom he deals. He must know his own stocks. He is held responsible for contamination. He must plan his work far enough ahead to prevent conflict. At all times he must carry in his head a picture of the jobs going on. This means not only pumpings that originate or come to his own department but must include pumpings on lines which pass through it and have connections on his own lines.

At night he is often called on to handle emergency repairs, or open clogged lines, often under the worst of conditions. He must watch tank temperatures, keep heavy oils in a condition to move and the lighter stocks warm enough that the water will settle out of them. Many stocks come to him wet or hazy. It is his responsibility to see that the water is drawn off and that nothing but dry oil is pumped. Batches must be mixed, and he is the man who is jumped on if by any chance they go wrong.

In addition to these and other responsibilities, he must record all movements of the stocks that come under his care. These are basic plant records from which costs and yields are taken, and they must be right.

The experience necessary to handle this work properly in a large pump house comes only after some years work as a helper. While this training is not acquired in night school it still is training and should be so regarded.

With this responsibility, it must be noted, goes a very considerable amount of hard physical work. The pumper must climb and gauge tanks in all kinds of weather. When a running tank fills up it must be moved regardless of wind, sleet, cats or dogs or skunks. And in all this the pumper must depend entirely on himself.

The foregoing is given not particularly to glorify the pumper but rather to stress the point that putting a man in charge of some new and fancy equipment does not necessarily mean that he has any greater responsibility, or that his work is of greater value than that of the man who may be working with little equipment on some of the less spectacular operations.

To begin with, the performance of a job survey must be taken seriously by all parties concerned. The management is interested in getting a check, not on theoretical or ideal conditions, not on conditions as someone would like to see them, but on conditions as they actually exist. The men should be interested in the betterment of working conditions as well as the increases in wages that many times come as a result of a job survey. The survey itself should not be allowed to become either a joke or a nuisance.

It is essential that the man making a survey in any department shall be thoroughly familiar with the operations of that department. This trite statement means exactly what it says. Too often office men are sent out to do this work. The job so far as they are concerned begins and ends with the filling out of certain set forms. Others go out and attempt to collect the information by kidding the boys along. They are perfectly satisfied to take what they get. Or a man may go out with his mind fully made up as to what a particular job covers. When he has checked his conception he is willing to call it quits and go no deeper. It must be kept firmly in mind that the purpose of a job survey, especially at this time, should be as much the checking up of conditions actually existing as the establishment of the regular duties and responsibilities of any particular job.

The information can best be gathered by questioning at least a couple of men on each job. The man on the survey should know the department well enough and spend sufficient time in it that he may select men capable of giving him the full information he needs. The questions should cover the smallest details of the work actually done. It is also well to get the foreman's idea of the job, and with this data the surveyor should send in his own interpretation. The idea of having every man fill out a questionnaire is not so good. To often the men most able to give real data will turn in the fewest answers. On the other hand, some rattlehead may turn in everything that comes into his mind and make a splendid showing. This method is especially unfair if an attempt is made to judge the capability of each man and his knowledge of his job from his questionnaire. The surveyor should have sufficient experience to put a real value on each detail and should send in his recommendations for the final specification for each job.

Further, the work of a department should be split up into its operating elements. The operation of each element should be studied and searched closely for any breakdowns in organization that work against real efficiency. Generally the trouble will come from some small irritation, poor planning that makes for unnecessary work, or the driving efforts of some petty strawboss to make a showing for himself. In most cases matters of this sort should be reported without too much of an attitude of placing blame. More often than not such slips in operation are a fault of the times, a hangover from the depression, and all too often the real fault may be high up in the organization, the foreman being forced to do the best he can with what is given him. Of course there are cases where the foreman is really not capable of handling his work or of holding the respect of his men, but here again some care must be used in attempting to remedy such a situation.

JOB SPECIFICATIONS

One of the first results of the survey should be the writing of job specifications. Just what is a job specification? First, it is a simple, clear statement of the duties and responsibilities of a man in performing the work assigned to him. It should outline not only the work to be done but also the manner of its performance. Why is it not good for the office or some supervisory group to lay out these specifications, to assign the duties and responsibilities without considering the actual performance or the man on the job? Why make a survey of it?

In every job, particularly in the types of operating jobs in a refinery, a thousand and one small items creep in which cannot be foreseen and which may not fit in with preconceived ideas of the job. The human element plays a greater part than it does in the planned operation of a machine tool. Proficient performance of the job usually requires a sense of organization rather than just manual dexterity. To be of any real value the specification must be based on things as they are and not on things as someone would like them to be. Every effort should be made to have the final specification lay out clearly the duties of the job without leaving a lot of loose ends. These specifications should be available to everyone so that each man may not only know what is ex-

pected of him but he will know what he must do to prepare for the jobs higher up.

Probably no part of the refinery organization has suffered more during the past few years than has the maintenance branch. In many plants the only maintenance work undertaken is that which has been forced. Just enough work has been done to fix breakdowns and keep necessary equipment moving. Equipment was shut down and left idle without being given proper care. The maintenance organization itself was often cut far beyond a safe minimum, and in many cases the cuts were not made wisely. Better class and higher paid men have been laid off, supervision has been cut to the limit, shops, tools and maintenance equipment have been allowed to run down. And worst of all, the maintenance organization may have lost the snap necessary for real results, may be shot through with small dissatisfactions that leave it dead, that make even the best men drift along, putting in the time and accomplishing as little as possible.

The efficiency of the maintenance department cannot be measured in the dollars and cents of the direct labor spent on it. Its effectiveness can only be judged from the way it keeps the plant equipment in condition to produce. And the cost of poor organization can more often than not be measured in terms of breakdowns, spills, low yields and lost production.

It is worth some pains to organize this department on the basis of preventive maintenance. There can be little argument on the fact that it pays to catch breakdowns before they occur, yet few plants make any real effort to organize with this purpose in view. The basis of such organization is the provision for regular inspection by capable, practical mechanics. This does not mean handing the job over to some cub just out of school who has not yet learned the difference between a gate valve and a monkey wrench. The men selected for such work should preferably be the older, thoroughly trained, all-around mechanics, men who really know repair work. They should make the rounds of the areas under their care at least once a month. A card record of each piece of equipment should be kept which shows the results of each regular inspection. From these inspections plans can be made to handle repairs before there is an actual break. By making arrangements with the operating foreman the work can be done without causing an undue loss of production. By cutting down on emergency jobs it is possible to have a tighter maintenance organization, and in the long run to carry less men, though in making cuts in working forces it must be kept in mind that the prime purpose of this department is to keep plant equipment in running order and that a few dollars shaved off the payroll may be over-run many times by even a small unnecessary loss of production.

In organizing maintenance work it is too often assumed that everything must be looked at from the viewpoint of emergencies, that it is not possible to do much planning. If an inspection program is carried out, by far the most of the necessary work can be as well planned as any other construction work. There must, of course, be a certain flexibility to the organization since breakdowns will occur. This combination of planning with a reasonable flexibility of organization spells efficiency in maintenance work. The survey should be directed toward such an effect. Here again the investigator should be experienced in this particular line of work. It especially is no job for a cub or a theorist. Only a man who has been through the mill, who knows the tribulations of maintenance work and who is a thorough mechanic himself can collect and pass on significant data along this line.

SALVAGED MATERIALS

This is also a good time to check over the spare and replacement parts on hand. It is easy to lose considerable time waiting for new parts to come. And what about salvaged parts? During the past few years equipment and parts were torn out and reinstalled without undergoing proper salvage operations and inspection. A surprising amount of real junk has been put back into hard service. That there have not been more serious breaks can only be attributed to the fact that the protection of the particular gods that look after drunks and fools must have been extended to cover many of our industrial plants.

And what about salvage policies and the salvage department itself. Few departments in the plant can be made to pay a better profit on the money invested in it than this department if it be properly operated. And it must be kept in mind that this profit comes almost entirely from materials that would be waste. It is a fact that much material is reclaimed and put back into service without undergoing any organized salvage. When a fitter needs a valve or a fitting he goes to some disused line, takes what he needs and installs it. The average valve reclaimed under such conditions is bound to leak slightly. Fittings put in in such a manner may be cracked, may be strained, may be badly corroded, or the threads may be in bad shape. A single break of such a fitting may cost far more than would many installations of new equipment. There is no reason why salvage operations, regardless of how small they may be, shall not be properly organized and carried out. It is not expensive to use proper care in taking out equipment so that it will not be damaged. It is not expensive to see that such equipment is properly inspected, repaired and tested before it is again put into service. The one rule here is: nothing goes back into service that does not test as good as new. In small refineries this may not require the establishment of a separate salvage department, but it will mean the establishment and carrying out of definite salvage policies.

The greatest losses in efficiency today come from breakdowns in organization. In many cases the very attempts to get more of a fancied efficiency have done little but bring about a gradual deterioration of the network upon which production hangs. In most plants during the last few years the working forces have been cut to a minimum or even below a minimum for safe operation. There are no men available to meet emergencies, and the operators are stretched to the limit to get the work done. It will be found that operating men are taking on work which could be done better and more cheaply by the maintenance department. It will be found that physical work and responsibilities are improperly distributed. It is points like this that a job survey can remedy. Orders may be given by staff men who accept little or no responsibility. Under these conditions department heads are apt to sit back and let matters take their course. If there is a jam, they are not holding the bag. Either the responsibility is passed on down to the operators or the tie-ups are accepted as a part of the day's work. Either way results in a don't-give-a-damn attitude that is bad for operation.

A Discussion of

Fractionation Practices

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EVER so often we have the pleasure of presenting one of those technical articles in non-technical style from the typewriter of J. A. Campbell. Often he disagrees with customary practice, and in this instance presents his disagreement with those who desire to pump cold reflux over the evaporator in the natural gasoline plant.

DURING the past few years there has been much discussion concerning the proper material to reflux over the top of the evaporator in an absorption gasoline plant. Some engineers take the stand that it makes no difference what is used, that the material pumped over the top of the column is only a cooling medium and is used merely to start the internal reflux down the column. Others claim that water will do just as well as gasoline.

Fractionation is an operation to which many factors contribute, and it is influenced by many laws. Consequently, it is thought, one particular theory can not be blindly followed. Discard fractions do not rise successively through the trays of a fractionator. Gasoline molecules entering the column at the feed plate may pass upward through several more plates in vapor form, be entrained and work back, be released and rise upward several more plates, and, after several throw-backs, finally reach the top of the column. Here some of the molecules may be condensed as reflux and be put back over the top plate and may work their way down through the fractionating section and even entirely down through the stripping section and out of the bottom with the lean oil. It is well known that the lean oil practically always contains some gasoline, usually a fraction of one percent, which is low but it is at least a showing.

Time and chance are also factors that enter into the scheme of things. It is known that water boils at 212°F. at atmospheric pressure. Try boiling a flask of water with a thermometer submerged and with another thermometer positioned so that its bulb is opposite the outlet. In watching both thermometers it will be noticed that they will remain at 212°F. so long as the lower one is submerged. When the water boils the first drop of condensate appears at 212°F. and considerably later the last drop appears at the same temperature. It might as well have been the first drop or any of the intermediate drops. Chance determines this. Time entered into the operation, because with a definite quantity of heat going into the water per unit of time, it required several units of time to put in enough heat to evaporate the water; this being termed latent heat. By observing closely it will be seen that some of the bubbles of steam liberated at the bottom of the flask never reach the surface. They are reabsorbed or condensed in the body of the liquid above.

To get down to cases, assume that a plant is operating a fractionating evaporator which is equipped with 10 stripping plates and 10 reflux wash plates. A 38 Baume kerosene distillate absorption oil is being used and operating practice requires that they take overhead from the evaporator, condense it in a cooling tower, and pump from the accumulator some of the resultant "raw" gasoline back over the twentieth plate of the evaporator. It is assumed that this reflux is at an average temperature of around 75°F. Enough of this reflux must be pumped back to cool the top of the column sufficiently to keep the end point within specifications. It should be remembered that we must distinguish clearly between temperature and heat quantity. A match will provide enough temperature to burst a high-range thermometer, but its heat quantity is not sufficient to perceptibly raise the temperature of a pint of water. What happens? A considerable quantity of this light gasoline pumped back over the column flashes off the top plate, passing directly to the condenser in vapor form. Moreover, the loss of latent heat from the top tray necessary to evaporate this large quantity of gasoline cools the top tray with the result that internal refluxing is set up. This, flowing down the column, cools off the rising vapor, and heavier reflux is condensed further down the column. However, a large amount of the outside reflux works its way down through the fractionating section and reaches the stripping section. When this descending reflux gets hot enough, other conditions being right, it flashes into vapor and takes heat quantity in the form of latent heat from that tray on which it flashes. Rapid heat flow causes a drop in heat pressure, which is temperature, so that the trays, progressively down the evaporator, are becoming cooler and cooler.

Thus the evaporator not only has the burden of normal gasoline carried into it on the tenth plate with the oil, but it has the overburden of all the external reflux and internal reflux thrown back from the stripping section. By this time the oil has become so chilled that it is necessary to supply extra heat by means of a reboiler or an intermediate heater, located somewhere below the feed plate.

Such a system requires a considerable quantity of steam to strip the oil, and requires large condenser surfaces to condense and cool the vapor recycle. Gasoline with a latent heat of around 130 is a very poor cooling medium as compared with water with a latent heat of 970. Even for sensible heat dissipation it is a poor medium, since its specific heat is less than half that of water.

A more effective and economical method of fractionation, according to this writer's opinion, consists of using a partial condenser outside the fractionator which should be water cooled. The partial condensate is of much lower gravity and it enters the top tray of the

column hot. This hot reflux washes back, for the most part, only the oil fractions and the heaviest gasoline fractions and it does not flash in large quantities, which flashing takes heat quantity in the form of latent heat from the apparatus. With this system the evaporator is not chilled and it is not necessary to provide a base or intermediate heater. The vapors will leave the top of the apparatus 80 to 100 degrees warmer than with the other system. If a box dephlegmator is used, this high-temperature heat is very readily dissipated by the evaporation of water. If the box is mounted on top of the cooling tower, the steam produced drifts off into the atmosphere and an important heat load is taken off of the cooling tower. The quantity of heat to be dissipated is very much less, because there is no supplementary heater; and because the temperature is so much higher the heat dissipation is much more effective through direct evaporation of water. A further saving is effected through utilizing the static head to flow the de-watered reflux back onto the top plate. No de-watering legs are necessary between the fractionating plates because the top temperature is too high to permit any condensation.

IDEAL REFLUX

It is a fundamental principle of fractionation that the ideal reflux is just one fraction lighter than the lightest fraction that the operator desires to drive out through the bottom of the column. This means that the reflux which should be used in the stripping section should be the heaviest fraction which can be incorporated in the finished product. Using a raw or even a stabilized gasoline is a direct violation of this principle and as well, is inefficient and costly. Using a cold reflux is apparently, an affront to common sense.

The writer used the hot reflux system in the first 45 gasoline plants built by his organization, and with excellent results. Many competent engineers so strenuously advocated the other system that in 1932, one of the other systems was installed in a newer plant. This change called for the doubling of the existing condenser coils which were of ample capacity under the old system. It called for adding six plates to the evaporator; for installing an intermediate de-watering tank, two large reflux pumps, one being a spare, and a large intermediate heater which was located four plates down from the point of feed charge. The heater at once became a source of trouble. This heater, of necessity, had to be designed for low-pressure drop, and the lack of turbulence caused it to scale up. Ever so often it was necessary to by-pass the heater and drill out the oil tubes, which operation added markedly to maintenance costs. When the heater was cut out for cleaning, 35 to 40 degrees were lost through the stripper. This permitted the oil to become supersaturated, and on starting up again often the evaporator would pop.

When hot weather came on the cooling tower was badly overloading, and the oil temperature was nine degrees hotter than under the former system. A spare cooling tower was available, which was reconditioned and into this was installed an additional 50 percent of condenser surface. This necessitated adding another water pump. The oil stripping was poor but more steam could not be used without raising the back-pressure on the evaporator to 50 pounds.

After two years of work with this expensive operation, a much shorter evaporator with eight stripping trays and nine reflux trays was built. A box dephlegmator was placed on top of the cooling tower utilizing the static head to return the reflux, thus eliminating the pump. The only changes in this system over the one which had been successfully used for years was the installation of a copper steam coil in each stripping tray and the use of nine reflux plates instead of four. In addition, a small de-watering leg was installed on each reflux tray, all connecting through seals with a common de-watering tank. The amount of water admitted to the dephlegmator box was controlled by the top temperature of the evaporator.

When this system was placed in operation it was found that the evaporator pressure was eight pounds lower, even when the condensers in the second cooling tower were removed from service. The full load was carried in one tower and the oil temperature dropped six degrees. It was found that the top temperature could be safely carried at 285°F. and still hold the end point of the gasoline below 320°F. Since there was no condensate in the boot-legs they were cut out of the system. Far better end point control, better oil stripping and better extraction were secured.

One thing, however, continued annoying the operators. The reflux was much heavier than under the system of not using auxiliary heat. It contained more absorption oil. The copper steam coils were effective, causing the oil to leave the bottom of the evaporator four degrees hotter than it entered the feed tray. Suspecting the coils of being responsible, the steam was cut off. Then it was found that the top temperature could be carried at 295°F. and that the total heat loss through the strippers was only 12 degrees despite the fact that extraction was proceeding at the rate of 5 gallons per thousand from the gas being treated. After making this change even better stripping was obtained, and practically perfect control of the end point was secured. The reflux became lighter by 4° Baume.

AUXILIARY HEAT NOT NECESSARY

In the light of this experience the writer is convinced that with a well designed stripping tray, auxiliary heat is not only unnecessary but may be decidedly disadvantageous. Putting heat in at the bottom or near the bottom of the stripper where the oil is fairly well stripped because of the monopolizing partial pressure effect of the steam, merely serves to vaporize absorption oil. These oil vapors rising through an increasingly hot zone reach the fractionating sections, and, because of the partial pressure effect of the gasoline vapors and fixed gas released on the feed plate, cannot condense. The result is that many heavy fractions that should not even reach the wash section, are carried out through the top in vapor form to overload the dephlegmator and interfere with its normal function. The removal of this unnecessary heat and the reflux pump, and the extra water pump has materially lessened the load on the plant boilers, saving considerable fuel, not to mention water.

Proponents of the finished-product-cold-reflux system often find it difficult to strip the oil. Various experiments are being carried out, such as using two evaporators, one operating on low pressure and pumping the oil to the second column. Superheated steam is being tried for agitation. Some are putting the oil into the evaporator at high temperatures, one plant using 370°F. However, the hotter the oil is handled, the more wild gasoline there is put into the top of the column to maintain end point control, and the more the top is chilled the more heat then is necessary to keep the light fractions from coming out at the bottom. After all, the evaporator is an "unloader," and certainly it is true that an efficient method for unloading is not one that calls for piling on.

Ammonia For Corrosion Control

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IN 1929 Walter Samans presented before a meeting of the American Petroleum Institute a report covering the cost to the oil refining industry of replacing and repairing corroded distillation equipment. He stated that a single tube still handling one million gallons of West Texas crude annually would require the expenditure of some $37,400 for repairs resulting solely from corrosion. Mr. Samans went on to say that the same still, with the proper injection of ammonia to control corrosion, would have required the expenditure of but $13,240 per year for both repairs and the ammonia consumed. This rather sizeable source of operating economy has not been overlooked by refinery engineers. Indeed, the use of various measures to control corrosion is becoming more and more a matter of necessity as an increasing proportion of sour and brine-contaminated crudes are being worked.

CAUSE OF CORROSION

Sulfur dioxide and hydrogen sulfide have been present in crude oil during distillation almost since the birth of the refining industry. It has been fortunate that normally the corrosion resulting from neither of these acid gases has been severe. In many cases a protective coating was formed which minimized the rate of corrosion. Only where rapid liquid streams eroded the metal under attack did the rate of corrosion assume major proportions. The use of alloy metal parts was relatively successful to check this type of loss.

Today by far the most serious source of corrosion of an acid nature results from the hydrolysis of magnesium chloride in crude oil when charged into distillation equipment. A sizeable proportion of the crude being worked today comes from wells yielding also magnesium brine. Attempts to completely remove magnesium chloride have so far met with scant success. It is safe to state that if the crude contains magnesium brine at the well there will still be a certain amount of magnesium chloride present when it reaches the still.

LOCATION OF CORROSION

Magnesium chloride in the presence of moisture will hydrolyze in the still at temperatures under 300°F. to form magnesium hydroxide and liberate hydrochloric acid gas which goes over with the distillate. Following the widely accepted theory, hydrochloric acid gas should not be corrosive at temperatures above the condensation point of moisture. Corrosion is seldom evident in the still or fractionating column. When corrosion in the fractionating column is reported, the reason frequently is that cold reflux liquid has caused localized water and acid condensation. Corrosion frequently takes place in heat exchangers and condensers where water and hydrochloric acid condense along with the distillate. As mentioned above, the use of alloy parts has provided a certain measure of relief but the cost of alloy equipment is such that it is generally not felt practical to use alloys except at the more critical points where mechanical erosion renders metal losses particularly acute.

ALKALIES FOR CORROSION CONTROL

For many years it has been common practice to add such alkalies as lime or caustic soda to the still along with crude oil being charged. These non-volatile alkalies afforded protection to the still itself and no doubt materially decreased the amount of acid gas leaving the still. Were it not for the hydrolysis of magnesium chloride it is entirely possible that the use of these non-volatile alkalies would be sufficient. In the case of hydrochloric acid it was found impossible to achieve even moderate degrees of neutralization by the addition of non-volatile alkalies.

Possibly as far back as 1920 refiners gave serious consideration to the use of volatile alkalies which could

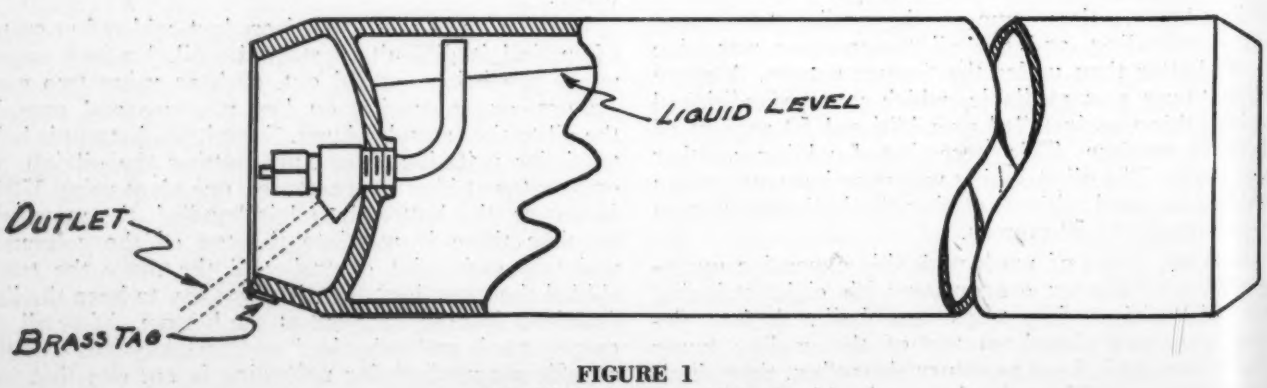


FIGURE 1
Anhydrous Ammonia Cylinder

be carried along with the distillate and afford protection at the point of initial water condensation. At present ammonia is the only suitable volatile alkali.

AMMONIA—THE CHEMICAL

Ammonia (NH_3) is, at room temperature and pressure, a colorless gas with a piercing odor. It is considerably lighter than air—at 70°F. and atmospheric pressure one pound of ammonia gas occupies a volume of about 22.5 cubic feet. Ammonia is commercially available in two forms—anhydrous ammonia, which is the dry gas compressed and liquefied into cylinders or tank cars, and 26° Baume aqua ammonia (also known as grade A ammoniacal liquor) which is a water solution of this same gas containing 29.4 percent NH_3.

SHIPPING CONTAINERS

Anhydrous ammonia is normally packaged in tubular steel cylinders containing either 100 or 150 pounds net of the liquefied gas and constructed to withstand a working pressure of 250 pounds per square inch. Cylinders are provided with an internal dipper pipe at the discharge end so that by properly placing the cylinder it will discharge either a gas or a liquid. Figure 1 shows a sectional view of an anhydrous ammonia cylinder in the correct position to discharge gas. It should be pointed out in this connection that at 70°F. a properly filled anhydrous ammonia cylinder will contain 85 percent by volume of liquefied ammonia and 15 percent by volume of open gas space to take care of expansion due to any normal temperature rises.

Anhydrous ammonia is also sold in 10,000-gallon tank cars containing approximately 52,000 pounds net. The tank car is in principle a large insulated ammonia cylinder mounted on trucks. Ammonia is ordinarily unloaded from the tank car into a 13,000-gallon pressure storage tank from which it may be removed either as a liquid or a gas.

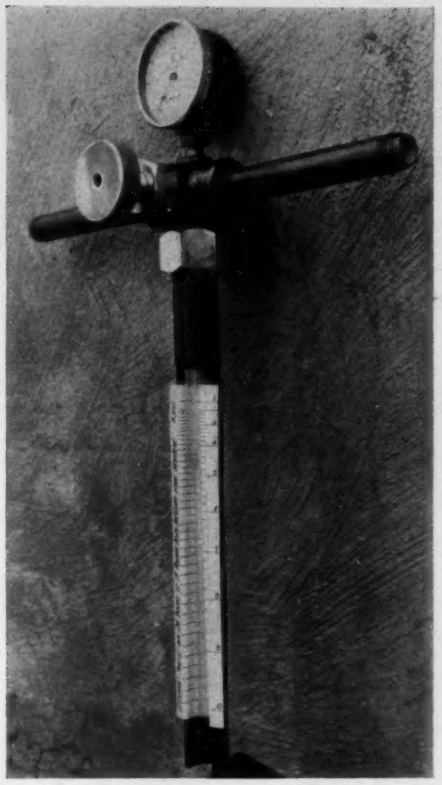


FIGURE 2

Flow Meter for Ammonia Gas

Photo Courtesy Pardee Engineering Company, Long Island City, N. Y.

Aqua ammonia (grade A ammoniacal liquor) is packaged in 55- and 110-gallon drums. These contain 380 and 760 pounds net respectively, equivalent to 111.8 and 223.6 pounds of NH_3. As the pressure exerted by aqua ammonia even under summer conditions is slight, aqua ammonia can be handled in a manner similar to that of any other volatile liquid.

Grade A ammoniacal liquor is also shipped in low-pressure tank cars. The ammonia may be withdrawn from the bottom of the car by a pump or gravity or blown out with compressed air. Later on in this article is a discussion of the proper method for tank car unloading.

AMMONIA COSTS

Under present market conditions anhydrous ammonia gas in 100- or 150-pound cylinders will cost about 16 cents per pound at destination, depending on the freight involved. The purity of this gas is in excess of 99.98 percent.

Anhydrous ammonia in tank car quantities is available in two grades —namely, refrigerating grade (over 99.95 percent purity) and commercial grade (over 99.5 percent purity). The refrigerating grade will cost about 5½ cents per pounds at destination depending on freights involved. Commercial grade anhydrous ammonia will cost about 5 cents per pound at destination depending on the freights involved.

Aqua ammonia of 26° Baume strength in 110-gallon

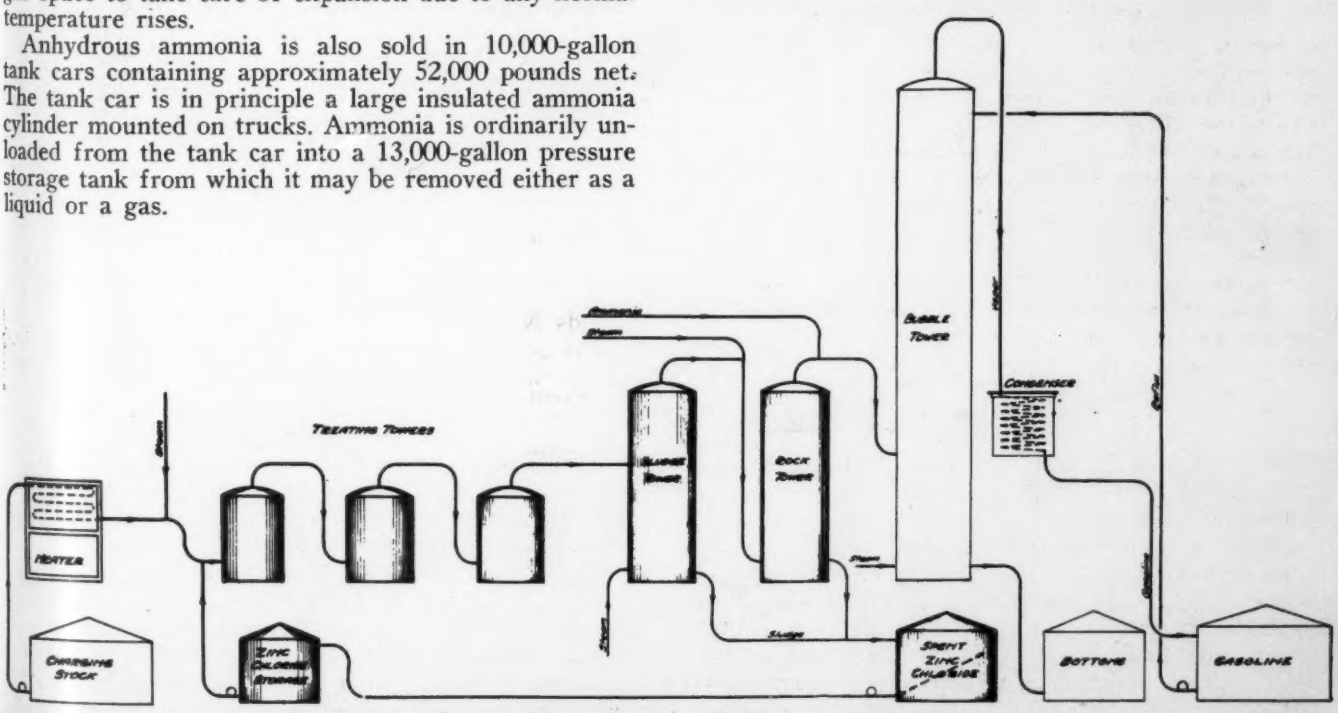


FIGURE 3

Application of Ammonia Gas to Lachman Zinc Chloride Treating Process

Drawing Courtesy of Vapor Treating Processes, Inc., Los Angeles, California

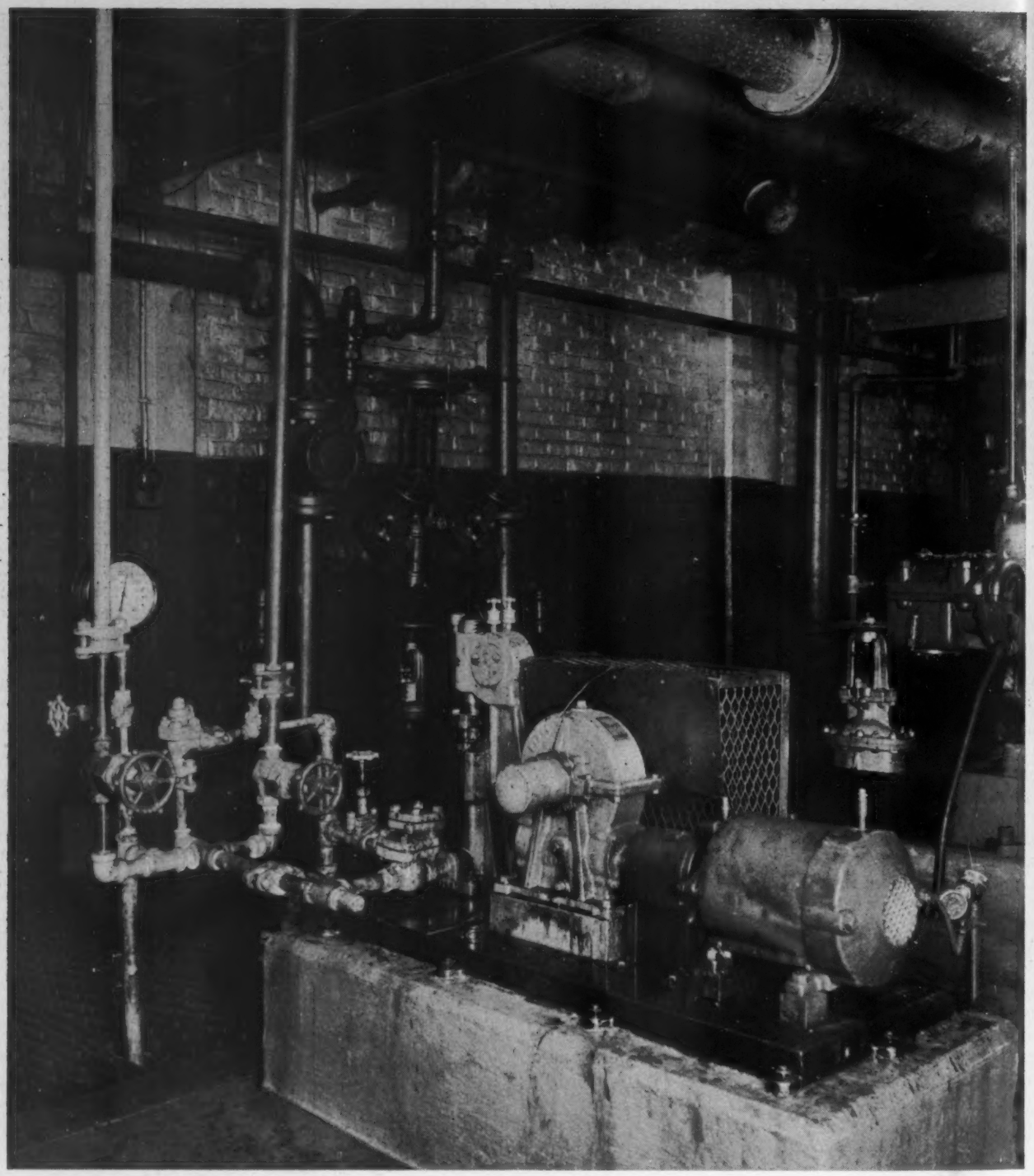


FIGURE 4
Motor Driven Pump Feeding Aqua Ammonia into Crude Charging Line
Photo Courtesy The Atlantic Refining Company

drums will cost between 2½ and about 3½ cents per pound depending on the distance from the ammonia producing plant. This price is equivalent to 8.5 and 12 cents per pound respectively of NH_3 content.

In 10,000-gallon tank cars holding 75,000 pounds net, (equivalent to 22,000 pounds NH_3) grade A ammoniacal liquor will cost around 1.5 cents per pound at destination which is equivalent to 5 cents per pound of NH_3 content. Here, also, transportation charges affect costs.

It is pertinent to state that normally a consumer taking less than the equivalent of one tank car of ammonia as anhydrous (50,000 pounds) or aqua (22,000 pounds NH_3) per year at any one point will find the use of cylinder anhydrous ammonia the cheapest. This statement takes into consideration labor, ammonia price and cost of injecting and storage equipment.

Consumers taking over 22,000 pounds of NH_3 at any one spot annually will frequently find tank car grade A ammoniacal liquor the most economical source of ammonia. However, in the consideration of tank car quantities a complication frequently arises in the fact that a refinery consuming this quantity of ammonia will often require anhydrous ammonia for refrigeration purposes. In such an event cost calculations frequently show that cylinder anhydrous should be used up to

50,000 pounds annual requirements and then a shift to tank car anhydrous ammonia is preferable.

PHYSICAL PROPERTIES OF AMMONIA

Table 1 indicates the more important physical properties of liquefied anhydrous ammonia at various temperatures.

TABLE 1
Properties of Anhydrous Ammonia

Temperature, ° F.	Vapor Pressure—Lbs. per Sq. In. Absolute	Volume of Compressed Gas—Cu. Ft. per Lb.	Density of Liquid—Lbs. per Cu. Ft.	Latent Heat in B.T.U.'s per Lb.
— 28	14.71	18.00	42.56	589.3
— 10	23.74	11.50	41.78	576.4
0	30.42	9.116	41.34	568.9
10	38.51	7.304	40.89	561.1
20	48.21	5.910	40.43	553.1
30	59.74	4.825	39.96	544.8
40	73.32	3.971	39.49	536.2
50	89.19	3.294	39.00	527.3
60	107.6	2.751	38.50	518.1
70	128.8	2.312	38.00	508.6
80	153.0	1.955	37.48	498.7
90	180.6	1.661	36.95	488.5
100	211.9	1.419	36.40	477.8
110	247.0	1.217	35.26	466.7

Table 2 shows the more important data applying to various concentrations of ammonia gas dissolved in water.

TABLE 2
Properties of Ammonia-Water Solutions

°Baume at 60° F.	Specific Gravity 60°/60° F.	Percent NH_3 by Weight	Gallons Water per Gal. 26° Aqua to Prepare	Freezing Point ° F.	Approx. Vapor Pressure—Lbs. per Sq. In. Absolute	
					40° F.	80° F.
10	1.0000	0.00	...	32.0	0.1	0.5
12	.9859	3.30	7.1	23.5	0.3	1.2
14	.9722	6.74	3.0	15.8	0.6	1.9
16	.9589	10.28	1.7	4.1	0.9	2.6
18	.9459	13.96	1.0	— 7.6	1.3	3.3
20	.9333	17.76	0.6	— 25.6	1.8	4.7
22	.9211	21.60	0.3	— 46.	2.6	7.2
24	.9091	25.48	0.1	— 71.	3.6	10.5
26	.8974	29.40	0.0	—105.	5.5	14.2

Ammonia is not a cumulatively toxic gas. Workmen have been engaged in the manufacture of ammonia over periods ranging up to 40 years and these men, though almost constantly breathing low ammonia concentrations, have suffered no ill effects.

TABLE 3
Physiologic Response to Various Concentrations of Ammonia in Air

	Parts per Million by Volume of NH_3
Least detectable odor	53
Least amount causing immediate irritation to eye	698
Least amount causing immediate irritation to the throat	408
Least amount causing coughing	1,720
Maximum concentration allowable for prolonged exposure	100
Maximum concentration allowable for short exposure (½–1 hour)	300– 500
Dangerous for even short exposure (½ hour)	2,500– 4,500
Rapidly fatal for short exposure	5,000–10,000

The pungent odor of ammonia serves as a warning agent and it is impossible to continue to inhale a dangerously high ammonia concentration.

In the event of an accident, ammonia liquid or gas might be sprayed over a workman and in general the treatment should be to flood affected areas with water and treat with mild acids such as vinegar or boric acid.

Theoretically, air containing between 16 and 27 percent ammonia by volume is flammable but these narrow limits of flammability are such that actually the combustion of pure ammonia seldom if ever is experienced at other than elevated temperatures. Ammonia mixed with oil will burn because the flame temperature of the oil is high enough to ignite and burn the ammonia. The addition of ammonia vapor to petroleum vapors does not increase their flammability but, if anything, decreases flammability. Although ammonia fires are sometimes reported in the refrigerating industry the cause of such fires is open to debate and evidence generally indicates that oil vapor mixed with ammonia supported the flame.

FIGURE 5
Detail of Connection for Injecting Ammonia into Crude Charging Line.
Photo Courtesy The Atlantic Refining Company

METERING AND PUMPING

When feeding anhydrous ammonia as a gas into refining equipment a simple method of control may be effected with a manometer type meter as shown in Figure 2. The ammonia flow is regulated by the differential needle valve as shown in the cut. Ammonia after going through the control valve passes through an orifice and the pressure drop across the orifice is shown in the calibrated manometer tube which hangs vertically. When using this type of meter it is possible to replace the orifice if the amount of gas to be handled falls outside the original calibration range. The discharge pressure of this meter is indicated by the gauge. Gas leaving the flow meter can be piped any distance, the pressure of the cylinder being normally considerably higher than the pressure required to force ammonia into distillation equipment.

Figure 3 shows how anhydrous ammonia as a gas may be added to prevent corrosion in a Lachman zinc chloride degumming unit. In this particular installation ammonia is added prior to the fractionating column.

Immediately prior to the point of ammonia injection into the column or vapor line an inverted **U** should be located. This type of construction prevents petroleum vapor from condensing in the cold line and running back into and clogging the flow meter.

Aqua ammonia must be pumped in an all-iron re-

ciprocating pump. Figure 4 shows an installation of this kind. Such pumps are frequently synchronized with the crude-injecting pump.

When feeding aqua ammonia it is sometimes desirable to add the ammonia directly to the crude oil prior to entering the still. Figure 5 shows the method in which ammonia is forced into the crude line. The center vertical pipe carries ammonia. The lower horizontal pipe carries crude.

Figure 6 shows the layout involved when feeding ammonia with the crude oil. Note that in this specific lay out full strength 26° Baume aqua ammonia is stored under ground and then blown with compressed air into a mixing tank where the concentration of ammonia is reduced to 19° Baume. An advantage of handling dilute ammonia lies in the fact that more accurate control may be effected when handling a larger bulk. Furthermore, the vapor pressure of dilute aqua ammonia is such that there is little probability of gas binding the pump in hot weather.

When the temperature of the still is below 900°F. the ammonia boils off along with petroleum vapors without loss. As still temperatures rise above 900°F. the ammonia will increasingly tend to dissociate in the still, giving nitrogen and hydrogen. Ammonia is not recommended for use in high-temperature equipment. In such units ammonia should be added at a point in the fractionating column where temperatures are only slightly above the condensing temperature of water.

pH CONTROL

As the purpose of adding ammonia is to give a neutral and therefore less corrosive condensate, the amount of ammonia added should be sufficient to increase the pH to about 7. Refiners generally use methyl orange, bromthymol blue or phenolphthalein as pH indicators. pH determinations are usually made on samples of water taken from the condensate. In the absence of sufficient water, a sample of oil may be shaken with water which will absorb the acid or alkali and give a relatively accurate pH reading.

The frequent use of methyl orange is probably accounted for by the fact that this same indicator is often used in oil refineries for other purposes. As it changes color in the neighborhood of pH 4, its use does not necessarily guarantee absence of corrosion. Frequently refinery operators are instructed to keep water condensate alkaline to methyl orange on the assumption that they will add sufficient ammonia to actually maintain a pH of slightly under 7. When methyl orange is used it is generally wise to check pH value with another indicator such as bromthymol blue to maintain condensate at nearer pH 7. From an operating standpoint the use of methyl orange frequently is most undesirable when water used to make the determination has a yellowish cast. Many Michigan oil refineries have found the use of methyl orange entirely unsatisfactory because the color change is masked by the color of the water itself. Michigan refiners have largely standardized on the use of bromthymol blue.

Bromthymol blue changes color at pH 7.0. On the acid side it is yellow. When alkaline the indicator is a bright blue. The color change is much more prominent than with methyl orange. Furthermore, bromthymol blue indicates acidity and alkalinity on the true theoretical basis.

The use of phenolphthalein is not popular but is used in a number of refineries where personnel are acquainted with it on account of its frequent utilization in the form of phenolphthalein impregnated paper for use in detecting ammonia leaks in refrigerating equipment. Phenolphthalein is colorless below pH 8. A pink color develops at higher values. When using all-iron equipment this slight excess alkalinity is not undesirable. It may cause the use of slightly excessive quantities of ammonia which from a cost standpoint is undesirable.

When Admiralty metal is used in condensing equipment refiners frequently make pH determina-

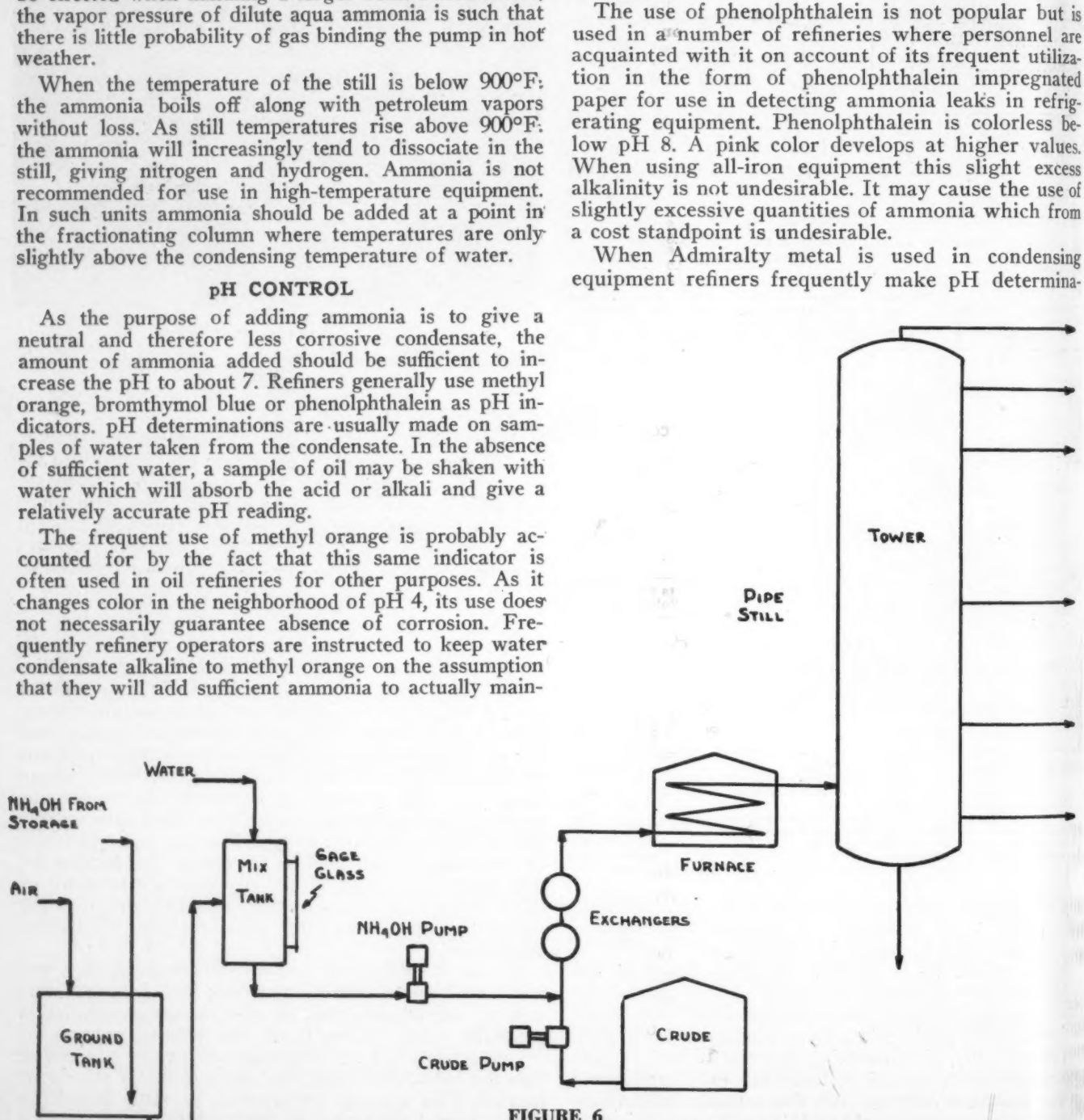


FIGURE 6

Flow Sheet Showing Method of Ammonia Addition to Crude Charging Line

Drawing Courtesy The Atlantic Refining Company

tions using both methyl orange and bromthymol blue. To effect a maximum of protection, water condensing with oil should be alkaline to methyl orange and only slightly acid to bromthymol blue, i.e. be just below pH 7.0. Admiralty metal should not be exposed to ammonia at much above pH 7.0.

Many refiners also determine iron content in condensate by using a colorimetric ferrocyanide test. A test of this kind is most convincing to the refiner who is not sure that the use of ammonia will pay for itself. When Admiralty metal is used in condensers it is sometimes desirable to also test the condensate for the presence of copper.

MATERIALS OF CONSTRUCTION

Refiners initiating the use of ammonia should be sure that all equipment for its handling is made of iron or steel. In the purchase of valves or unions special care must be taken to avoid the use of so-called "all-iron" units which contain copper alloy seats.

Gauges should be fitted with steel tubes. Air gauges are almost always fitted with brass tubes and are therefore, unsuited for ammonia service.

Tanks for handling aqua ammonia of any concentration should be of all-iron construction and should be tested to 25 pounds gauge. Such tanks should be fitted with a safety relief valve set to open at 25 pounds. Such a valve will open only in the event of filling the tank without venting it. In this case any air overlying the liquid ammonia will be compressed and generate considerable pressure.

Oddly enough, the chief concern is to protect the tank against vacuum. In hot weather the gas overlying aqua ammonia will be largely ammonia fumes. In the event of sudden cooling, as during a thunder storm, this ammonia will suddenly condense, pulling a vacuum. A large size vacuum breaker should be installed to open at about 5 inches of water suction. Such a vacuum breaker can be spring-loaded or simply be a water seal which will suck air. (The same water seal will protect against excessive positive pressures.) Care must be taken that the water seal never becomes plugged from rust or other foreign material deposits. In winter, water seals should be protected against freezing by the addition of a suitable anti-freeze. Frequently ammonia gas dissolved from the storage tank will give adequate protection. Irrespective of how freezing is guarded against, hydrometer readings should be taken to assure the maintenance of the proper concentration of anti-freeze. It is an interesting fact that ammonia tends to prevent any rusting on the part of iron.

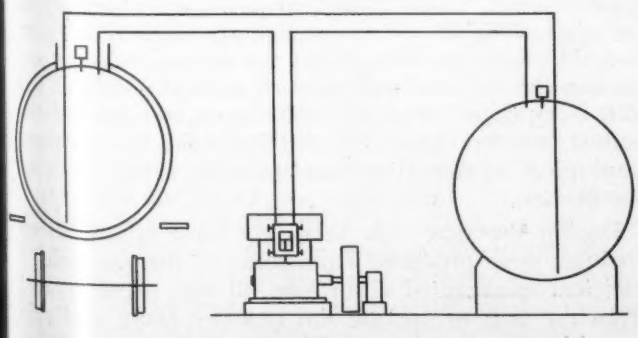


FIGURE 8
Anhydrous Ammonia Car Unloading Lay-out Showing Tank Car, Gas Compressor and Receiver.

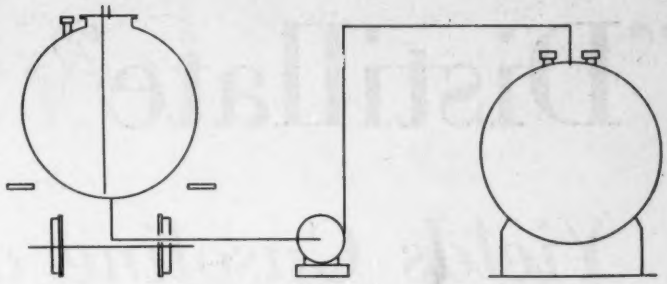


FIGURE 7
Ammoniacal Liquor Storage Lay-out.

However, a tank or piping which has contained ammonia should never be exposed to moist air in the absence of ammonia vapor. If this happens severe rusting will ensue.

Figure 7 shows a typical lay out for the emptying of tank car quantities of aqua ammonia to storage. This particular lay-out shows a centrifugal pump taking aqua from the bottom of the car and discharging it into a storage tank. Cars may also be unloaded by gravity as is the case in the lay out shown in Figure 6. Under some circumstances it is desirable to unload the car by applying low pressure air above the liquid in the car. This procedure is not recommended because of the danger of applying too high a pressure.

Figure 8 diagrammatically illustrates the lay out for unloading anhydrous ammonia tank cars. The principle of tank car unloading is to create a continuous pressure differential between the two tanks by sucking ammonia gas from the storage tank and compressing it over liquid in the tank car. Liquid anhydrous ammonia is then blown over. On account of its high vapor pressure the pumping of liquid anhydrous ammonia with centrifugal or reciprocating equipment is not practical. The handling of tank car quantities of anhydrous ammonia requires special equipment whose detailed description is beyond the scope of this paper.

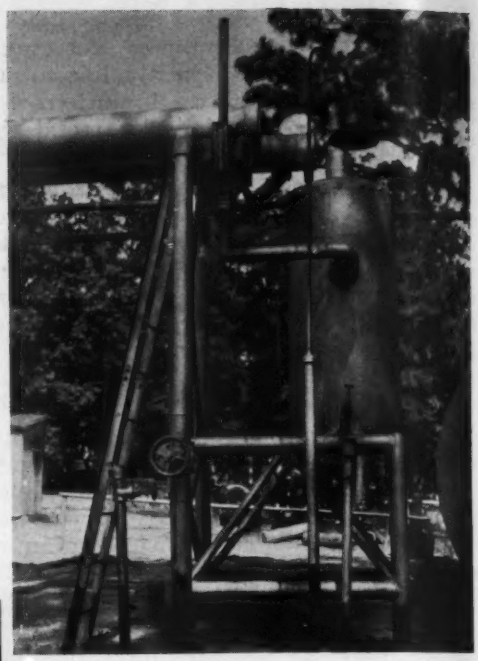
Pressure relief valves should be provided wherever there is any danger of building up excessive anhydrous ammonia pressures. For the protection of small equipment a spring-loaded relief valve set to open at 250 pounds is recommended. It should be borne in mind that valves of this type frequently are not entirely ammonia tight. Also after opening they may not seat tightly. For this reason relief valve assemblies for protecting equipment such as large storage tanks provide rupture discs both in series and parallel with spring-loaded valves. Furthermore, such units are always provided in duplicate.

CONCLUSION

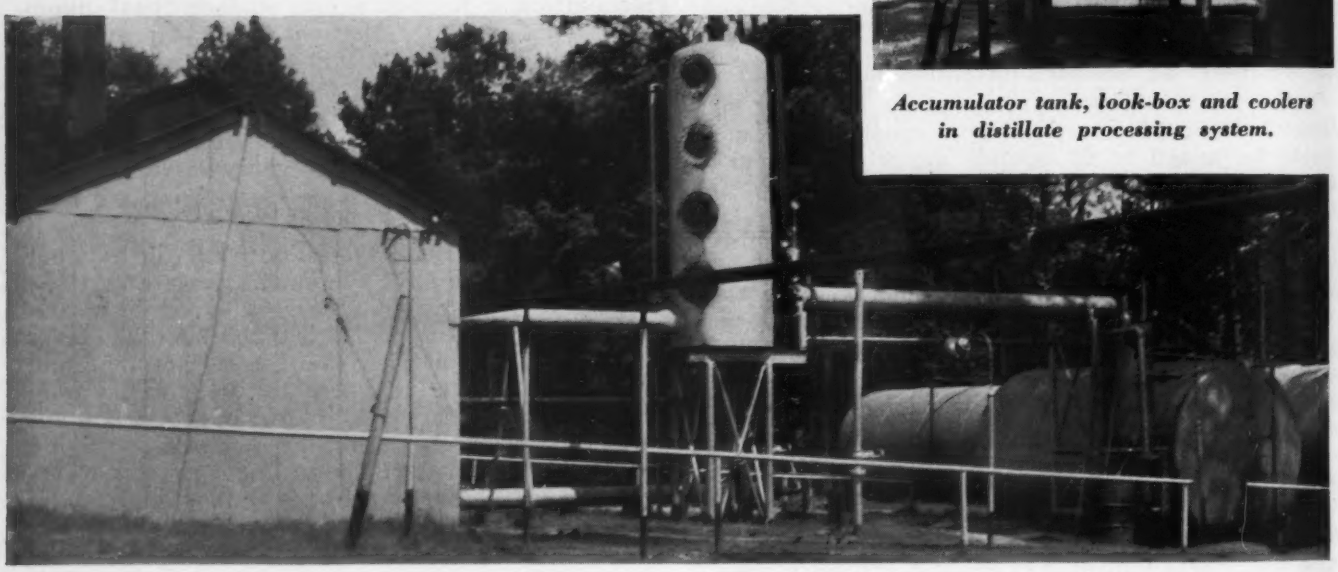
Experience indicates that ammonia in its role of a volatile alkali will continue to play a prominent part in oil refining operations. With the development of new petroleum products and advanced refining technique, ammonia may find new points of application both as an alkali and as a refrigerant. The foregoing paper is not intended to give complete details concerning either the handling of ammonia or its proper application. As specific ammonia problems vary with local conditions, each refiner's ammonia problems require individual consideration.

"Distillate"

Yields Gasoline and Absorption Oil



Accumulator tank, look-box and coolers in distillate processing system.



Bubble column which is used by Arkansas Fuel Oil Company to fractionate "distillate" from gas wells in the Rodessa, Louisiana field into gasoline and absorption oil. The still is housed in the building at the left.

ARKANSAS Fuel Oil Company handles "distillate" from several of its gas wells located in the Rodessa field by processing it in the gasoline plant. Only a few of the wells connected to this company's system of gas-gathering lines produce this light, high-gravity oil, but quantities have been sufficient to justify installation of special processing equipment.

This distillate is handled through separators and traps in the field in the same manner as the heavier crude. It is pumped into storage located at the gasoline plant and subsequently distilled to produce two products; the lighter overhead fraction having an end point sufficiently low that it may be included in the normal gasoline production, while the bottoms, or heavier fraction taken from the base of the bubble tower has boiling characteristics similar to the usual grade of absorption oil.

The equipment used for this purpose was accumulated at the plant, and consists principally of idle material not in use in the regular absorption system. The still used for the purpose of boiling the charge was made from an oil field boiler. Pumps were installed to circulate the charge and to feed fresh oil to the system. A four-plate bubble tower was erected to fractionate the charge which is controlled by refluxing the overhead section with condensate. The vapor line leads from the top of the bubble tower and is connected to a horizontal tubular water-cooled condenser.

A vertical accumulator tank at the outlet of the condenser section dumps its charge into the regular gasoline stock tanks. Temperatures are maintained on the still and bubble column sufficiently high to drive the desirable light fractions from the charge, without exceeding the required end point of natural gasoline. The still is equipped with a Dutch oven, set behind the firebox, so the flames can distribute the heat without impinging against the flue sheet or other parts of the firebox.

During the time that relatively large quantities of distillate were produced from some of the gas wells, a sufficient quantity of absorption oil was manufactured from the unit to operate the gasoline plant, and ship quantities to other plants. The operation of the rerun unit did not require additional employes, since automatic equipment was installed for control.

Liquid-Level Indicator Set-Ups

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The Meriam Company, Cleveland, Ohio

THE need for knowing accurately the level of liquids in tanks exists in practically every industry, whether it is an important function in actual manufacturing processes or whether it pertains entirely to such equipment as fire protection and sprinkler installations. Liquid-level instruments may merely indicate the conditions existing or they may be so designed that they also control the pumps which maintain that level. They may be of the Bourdon or dial type, or of the manometer kind. Details given below refer to the well type manometer instruments, since these are not only usually less expensive but also less liable to inaccuracy or to errors due to wear.

The basic principle of the manometer when used as a tank gauge is that the pressure, due to the head of liquid in the tank, is transferred to the liquid in the well of the manometer and this pressure is balanced by the rise of the gauge fluid in the manometer. In some cases it is advisable to have the manometer mounted at the bottom of the tank and the liquid pressure conected directly under the fluid in the manometer well. In other cases it is advisable to mount the instrument at some distance from the tank, and in this case it is necessary to run a line from the well of the manometer through the top of the tank, into the bottom of the tank. Air is then forced into this line from the manometer to the bottom of the tank, and as soon as the air pressure equals the pressure of the liquid head in the tank, the air will bubble out and canot exceed the pressure of the liquid in the tank. Inasmuch as the air pressure is the same in all parts of this line, this air pressure also acts upon the fluid in the manometer, causing the fluid to rise in the tube of the manometer until it just balances this air pressure. If both the liquid in the tank and the liquid in the manometer are of the same gravity, the instrument level will rise to the same height as that in the tank. If the specific gravities of the liquids differ, as is usually the case, the relative heights will be inversely as their specific gravities. Thus a mercury-filled manometer measuring water-tank level will show a rise of only about 1/14 of the water height, mercury being approximately 13.6 times as heavy as water.

The manometer scale is, of course, calibrated accordingly, or if the dimensions of the tank are known it can be calibrated equally well to read in gallons or other convenient units.

To determine the length of manometer required, the following very simple formula is used:

$$h = \frac{H \times SpGT}{SpGM}$$

h = scale length of the manometer in inches
H = fluid depth in the tank in inches
SpGT = spec. gravity of the fluid in the tank with respect to water.
SpGM = spec. gravity of the meter fluid.

The accompanying illustrations show diagrammatically the installations of manometers on open

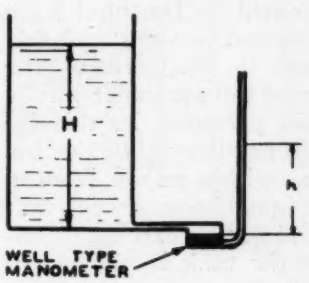


FIGURE 1

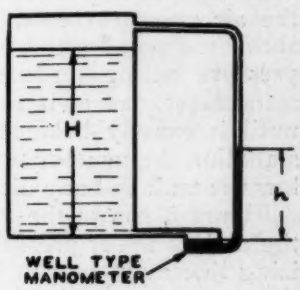


FIGURE 2

and closed tanks. Figure 1 shows the installation of the manometer on an open tank where the fluid is directly on top of the fluid in the well of the manometer. Since the top of the tank and the top of the manometer are both open to atmospheric pressure, there is no need of any connection to the top of the tank. On this installation, the distance H and h vary inversely as the specific gravities of the fluid in the tank and the fluid in the manometer.

Figure 2 shows the same installation except that the tank is closed and pressure may exist on top of the fluid in the tank. In this case an equalizing line is necessary so that the same pressure is on top of

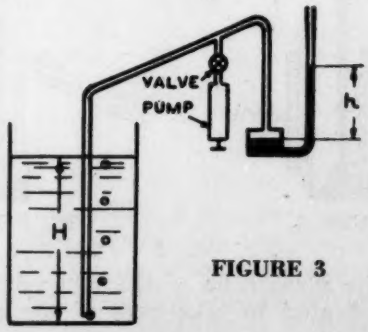


FIGURE 3

the fluid in the gauge and the fluid in the manometer, which therefore rises and falls in exact accordance with the rise and fall of the fluid in the tank.

Figure 3 shows a manometer installation where the manometer can be located at any distance from the tank. In this installation the line runs from the top of the tank to the bottom of the tank and over to the well of the manometer. As the fluid rises in the tank, it also tends to rise in the line leading to the bottom of the tank. This compresses the air in this line and causes the fluid to rise in the manometer.

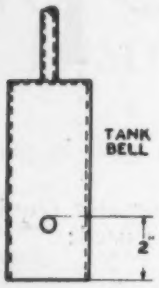


FIGURE 4

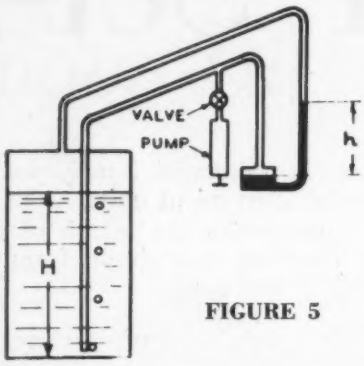


FIGURE 5

To obtain correct readings, it is necessary to introduce air until the air bubbles out of the line at the bottom of the tank. This may be done by a hand pump as shown on the diagram. When the air is bubbling out of the line at the bottom of the tank, the air pressure is exactly equal to the fluid head above the point where the air bubbles out and this pressure acting on the fluid in the well of the manometer, causes it to rise in the manometer tube until it exactly balances this pressure. In this installation the manometer may be either above or below the tank and any distance, within reason, from it.

Figure 5 shows the same installation except that the tank is under pressure and in this case an equalizing line must be run from the tank to the top of the manometer tube in a similar manner to that shown on Figure 2.

Figures 3 and 5 require the use of a valve, check valve and pump. To get away from this complication, air is introduced into the line leading to the bottom of the tank through the liquid in the well of the manometer. This is shown diagrammatically in Figure 6. By the use of an aspirator bulb, air is forced through the liquid in the well of the manometer, and upon releasing the bulb the fluid rises in this line leading to the aspirator bulb and effectively prevents any loss of air pressure by this liquid seal. The liquid will rise in this line the same height as

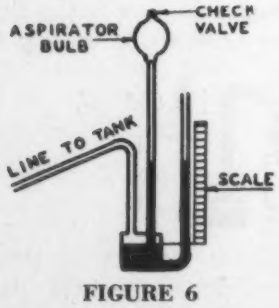


FIGURE 6

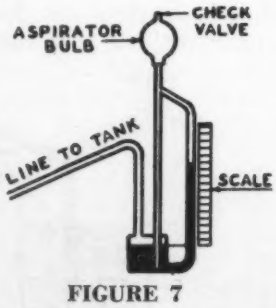


FIGURE 7

shown in the glass tube of the manometer and the scale is calibrated to take care of the drop in the well as the fluid rises in both of these tubes. On Figure 7 is shown a by-pass from just below the aspirator bulb to the top of the manometer tube. The purpose of this by-pass is to prevent the fluid being blown out of the gauge by introducing air too fast into the well of the manometer, before it is equalized in the line leading to the bottom of the tank; for in such a case it may build up sufficient pressure to do this. By using this equalizing line, the liquid is depressed in the glass tube of the manometer when the bulb is squeezed, and cannot be blown out. Figure 8 shows this manometer with the aspirator bulb on the tank under pressure. This is for use on storage tanks such as used for gasoline or naphtha where the vapor pressure is only a few ounces. In operating the gauge it is necessary to squeeze the bulb, open the valve and force air through the liquid in the well of the manometer. Then the valve is closed and more air is introduced into the pump, and the operation repeated until the liquid stops rising in the tube of the manometer.

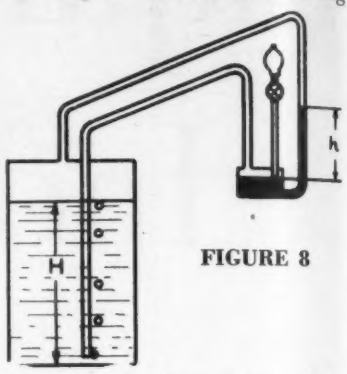


FIGURE 8

Figure 9 shows the same type of installation except that moisture traps are provided just ahead of

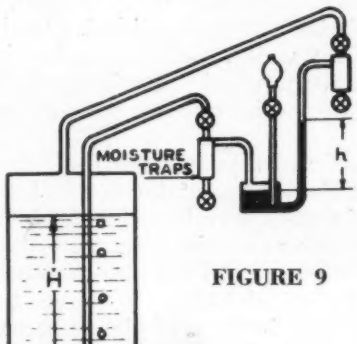


FIGURE 9

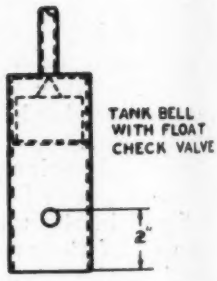


FIGURE 10

the manometer. This is used where the liquid in the tank may be quite volatile and may condense at atmospheric temperatures. These moisture traps are for the purpose of keeping the condensed fluids out of the gauge itself.

Wherever the pressure on top of the fluid in the tank may be 5 or 10 pounds pressure, it is recommended that a sight feed bubbler as shown on Figure 13 be used.

Where this sight feed bubbler is used, it is necessary to run an air line, having approximately 15 pounds pressure, up to the bubbler. This is shown diagrammatically on Figure 15. Due to the fact that practically all tanks collect sediment and dirt in the bottom, it is advisable that the first 2 inches of fluid be not measured. A tank bell is usually fastened to the line leading to the bottom of the tank and this bell has openings, the top of which comes 2 inches from the bottom of the bell. The air bubbles out through these openings and consequently the first 2 inches in the tank are not measured by the gauge but the scale reading starts at 2 inches and reads up.

Figure 10 shows such a tank bell with a float check valve. If the air pressure should fall in the

line as the liquid would tend to rise in the tube, it lifts this check valve off its seat and closes off the line. This may be desirable on some installations.

On Figure 11 is shown an installation where the manometer is some distance below the bottom of the tank and the connection is made from the bottom of the tank to the well of the manometer. In this case it is necessary to correct for the additional head D by raising the zero point of the scale a distance on the manometer; otherwise the installation is the same as shown on Figure 1.

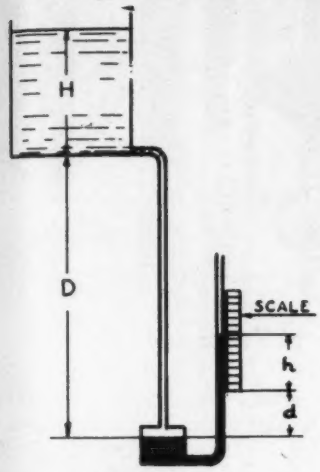


FIGURE 11

On some installations of tanks under pressure, it is desirable to mount the manometer at the bottom of the tank and to have the same fluid as that in the tank on top of the gauge fluid in the glass tube of the manometer. Figure 12 shows such an installation and here a seal pot is used at the top of the tank and connection made from this seal pot to the top of the tube on the manometer. This seal pot is then filled with the same fluid as in the tank and condensation of the fluid in the tank tends to keep the seal pot filled at all times. It will be noticed on this installation that the well of the manometer has been placed at the top and not at the bottom as shown in previous installations so that the zero on the scale is at the bottom. As the tank fills up and the level in the tank approaches the level of the seal pot, the fluid in the tube of the manometer rises toward the full mark on the scale.

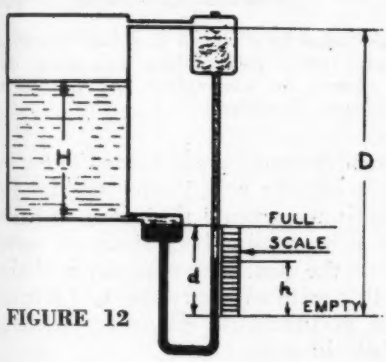


FIGURE 12

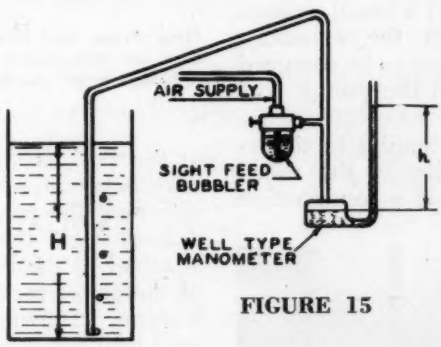


FIGURE 15

Figure 14 shows a mercury seal between the fluid in the tank and the manometer gauge. This installation would be used on such material as naphthalene, which becomes solid at ordinary temperatures and must be heated in order to become liquid. The mercury seal balances the pressure of the fluid in the tank and by running a line from the well of the manometer through the mercury in one leg of the mercury seal, the gauge can be calibrated in terms of height H of the fluid in the tank.

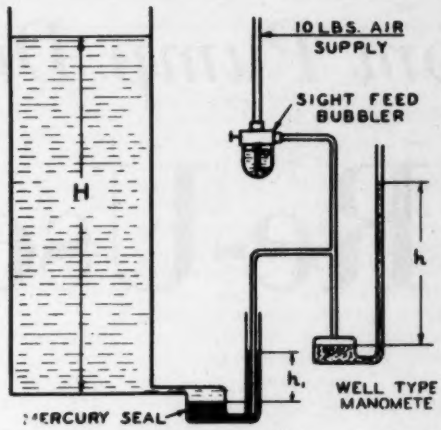


FIGURE 14

Figure 15 shows the installation of the sight feed bubbler where it is desired to bubble air continuously into the line leading to the bottom of the tank. In the event the tank is under pressure, it is therefore necessary to have an equalizing line from the top of the manometer to the top of the tank.

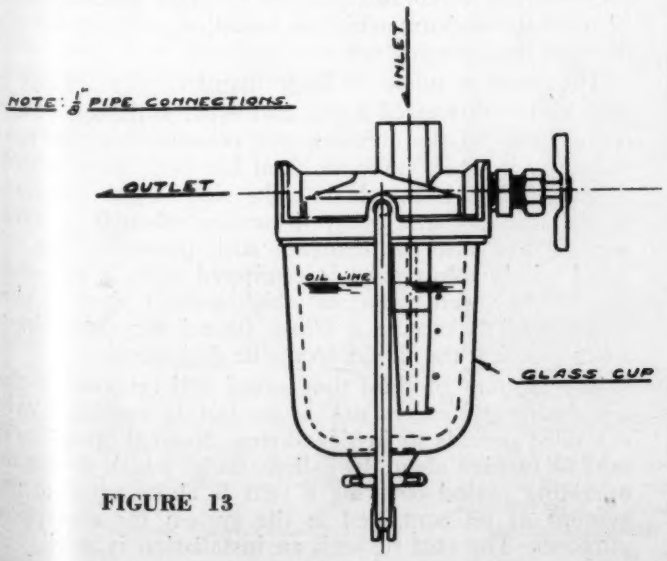


FIGURE 13

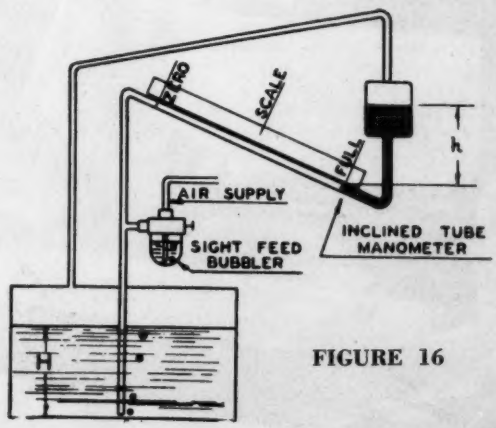
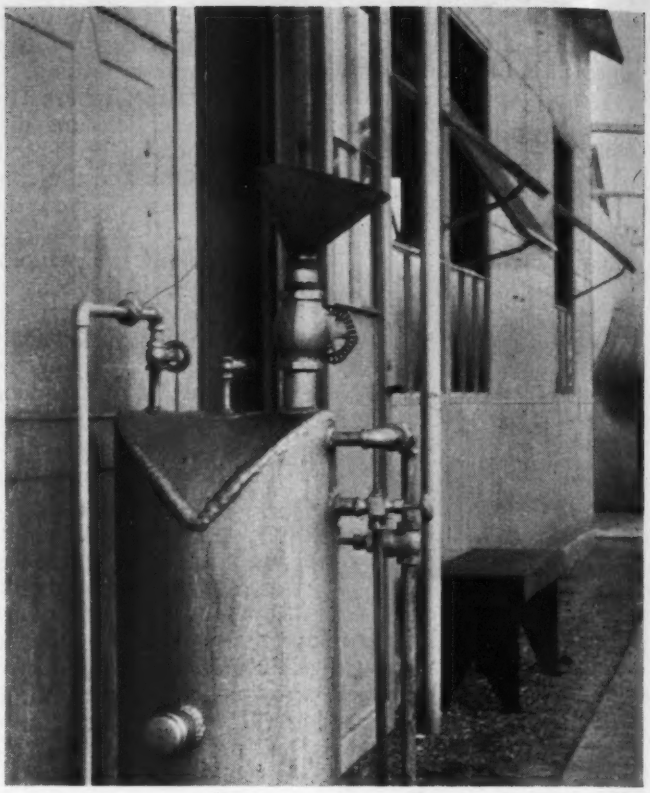


FIGURE 16

Figure 16 shows an installation on a tank under pressure where a sight feed bubbler is used and one leg of the manometer is inclined at an angle to give a longer scale reading for the displacement height of the fluid in the manometer. These diagrams cover practically every installation encountered in the use of the manometer as a liquid level indicator.

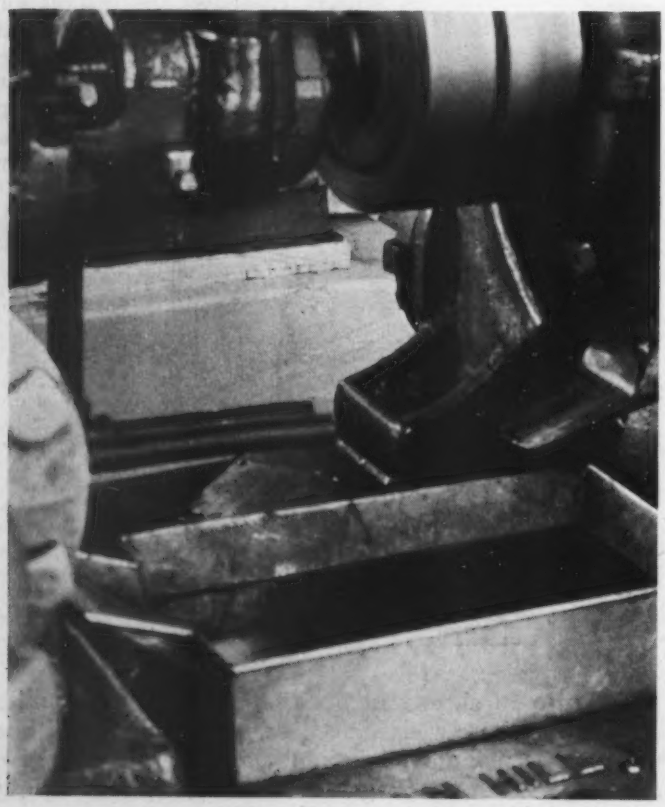
Oil *from Pump Drips* Re-Used



Drip drum and blow-case used by Coltexo Gasoline Corporation in conjunction with drip pans when salvaging oils leaking past packing glands on absorption oil pumps at Rodessa, Louisiana.

USING high-speed turbine-driven centrifugal pumps for rich and lean oil circulation, Coltexo Gasoline Corporation, Rodessa, Louisiana, does not desire to hold pump shaft packing sufficiently tight to reduce all leakage past the packing gland and stuffing box. It has been found that packing will give better service and provide longer life to the installation if a small amount of fluid is allowed to leak through the assembly. Naturally this condition caused drip pans to be designed and placed on the bedplates between the pumps and the steam turbines.

Pans designed by this company are similar to single-loaf bakery pans, but have in addition to the rolled rim, a lip, or spout at one end which is a continuation of the stock from which the pans were made. The wire reinforcement which is usually rolled into the rim for stiffening the pans continues around the spout to make it more rigid than if it were simply a sheet of metal attached to the body of the pan. The capacity and size of the pans are such that with ordinary use and normal leakage, removing the accumulated oil occurs perhaps each tour, or twice daily in some cases.



Drip pans beneath centrifugal pump and steam turbine packing glands to catch drips so the oils may be saved for reuse. Coltexo Gasoline Corporation, Rodessa, Louisiana.

Mineral seal oil, or absorption medium permitted to leak through the packing has in addition, condensed steam that normally drips from the packing of the steam turbine. Such contamination is not considered as unsatisfactory performance, and each pan catches the accumulation of both oil and water. To salvage oil thus permitted to escape through the packing, the pans are removed when full and the contents poured into a blow-case, or drum which is installed just outside the door of the pump house.

The drum is made of large diameter pipe, closed at each end with welded heads and fitted with nipples and connections so that residue gas pressure may be used to empty it when sufficient fluid has been accumulated. A gauge glass is attached to the side of the blow-case so that the operators may determine when it is necessary to evacuate the contents with pressure. The top head of the blow-case is equipped with a two-inch nipple and a gate valve, in which a short piece of pipe is screwed to receive a filling funnel for convenience when pouring the liquid from the drip pans.

The amount of fluid thus saved and returned to the circulating system is not large, but is sufficient over extended periods to justify saving. Normal operation is said to furnish about 10 gallons daily, which during an operating period covering a year is about equal to the amount of oil contained in the system for absorption purposes. The cost of such an installation is small.

IDEAS to help plant efficiency

Blow-down & Drop-out Line Supports and Retaining Fixtures

WHEN laying the cracking plant blow-down and drop-out line to the receiving tank, seamless pipe was used by Black Gold Refining Company, Oklahoma City, welded at the joints and placed

Blow-down and drop-out line supports and retaining slides installed on cracking plant.

upon pipe supports to prevent contact with the soil. Instead of the long and costly expansion bends, this line was laid with a long, easy turn to compensate for radical changes in temperature when in use. Where the pipe was laid in a straight line near the tube stills and pressure vessels, the pipe rested upon cross members without any retaining devices. At the point of the bend, two extra long supports were placed for the pipe to rest upon, yet providing sufficient surface for the sliding action which the pipe takes when hot oil is flowing through it. Retaining bars were installed above the pipe and supports, securely bolted to the slides to prevent vertical movement of the blow-down line.

Portable Tank Cleaner

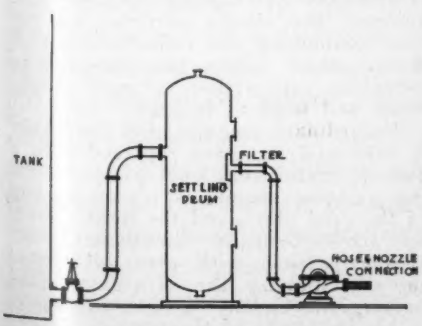
HEAVY oil tanks often become dirty due to collection of silt, coke particles and basic sediment, and this, of course, necessitates cleaning which is often an expensive procedure.

The accompanying sketch shows one system of tank cleaning apparatus. A suction line from the tank to be cleaned, a settling drum, filter, and circulating pump comprises the system. The tank is pumped down to a point as low as possible, but leaving sufficient liquid to get suction on the outlet.

Contents of the tank are circulated through the settling drum by means of the pump. It is transported back to the tank top manhole through a hose and nozzle which is connected to the pump discharge. The nozzle agitates the tank settlings and hastens cleaning.

In order to start circulation sometimes the system must first be filled with water or oil. If the tank liquid is too viscous a steam jet may be used to create a pull on the tank. The apparatus can be mounted on any suitable means of conveyance and when the drum is filled up with sediment the drum can be emptied at any convenient or suitable location. The filter on the drum discharge permits all settling to stay in the drum during cleaning.

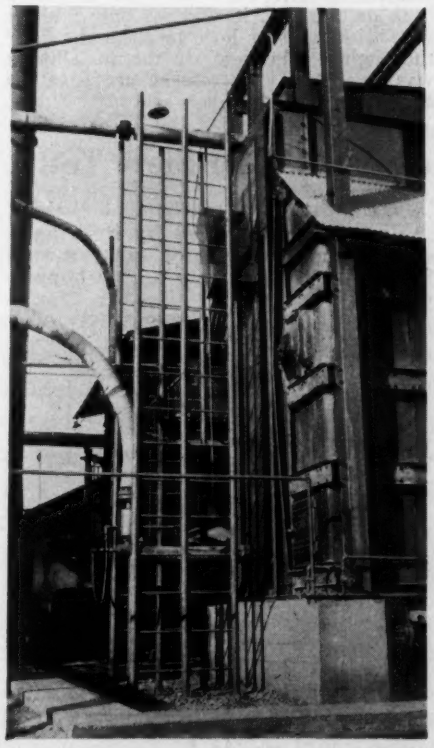
M. E. WOOLDRIDGE,
Ponca City, Oklahoma.



Portable Tank Cleaner

Use Cracking Still Ladders for Scaffolding Supports

WHEN the heavy oil pipe still was built in the plant of the Black Gold Refining Company at Oklahoma City, ladders were placed at each end of the furnace where the tube headers are exposed when turbining tubes. The ladders are made by the plant welder, using two-inch pipe for the legs and short lengths of sucker rods for the rungs. Each ladder has three legs, making a double ladder on each side of opening which exposes the tube headers. The rungs are spaced for convenient climbing to the top of the tube still, and also spaced so that scaffolding can be used when the workmen are operating the tube cleaning equipment. With this method of a combination of ladders and scaffolding supports, planks may be placed at any point from just above ground level to the top of the heater. Elimination of fire hazards is accomplished by using metallic ladders, and safety is promoted, also, by the permanence of the equipment. The cost is negligible and the installation does not detract from the appearance of the unit.



Ladders built at end of heavy oil furnace in cracking plant, so that they may also be used as supports for scaffolding when turbining tubes of heater.

Reconditioning of Cylinder Graduates

CYLINDER graduates with broken tops, test tubes, and other glass ware with cylindrical shape may be quickly repaired. Graduates with large diameters are very difficult to cut off by using glass tubing cutters, but they may be easily and quickly cut along a perpendicular cross section by the use of a hot wire.

The best results are obtained by using size 18 copper wire. The wire should be bent in the shape of a half hitch with the loop slightly larger than the cylinder. A right handed person should hold the graduate in the left hand with the broken end on the thumb side of the hand. Place the loop of the wire around the graduate and bend one free end so that it will lay along the side of the cylinder and may be held in place with the left hand. The other free end of the wire should be longer than the circumference of the graduate. Hold the extreme end of the free wire with the right hand while passing the wire between this hand and the graduate over a flame until it is heated red. As quickly as possible after this is done, with a circular motion of the right hand, wind the hot wire around the graduate using the loop of the half hatch as a guide. Hold the hot wire in this position for about 30 seconds; then quickly remove the wire and submerge the hot end of the graduate in an ice-water bath. The end of the graduate will break off smoothly along the circumference where the hot wire had lain. After freeing the graduate from water, the sharp edge may be smoothed by burnishing.

The repair may be made by another method with more speed which would be especially useful to a firm that has a great number of graduates or cylinders to repair. The copper wire may be replaced by a resistance wire which should be of such resistance that it will become heated red when connected to the 110-volt light circuit. The wire should be wound one com-

plete turn at the desired place of cutting the cylinder; then after heating the wire by passing a current through it the above procedure of chilling will cleanly cut the glass.

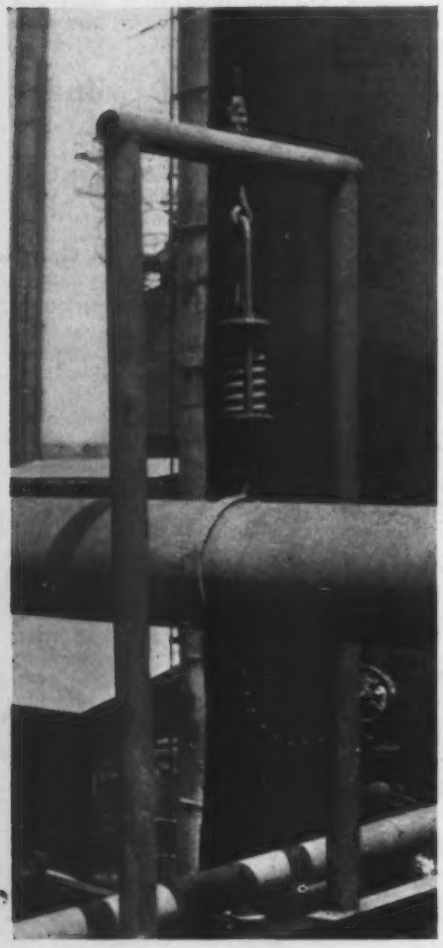
A spout, if desired, may be easily bent into the graduate at the time of burnishing by the use of a small iron rod. While the top of the graduate is heated cherry red, the spout may be formed successfully by pressing the heated, but not red, rod against the inside of the cylinder and continuing to do so until the glass has taken the desired form; then remove the rod and allow the glass to cool. Great care must be taken to not allow the iron to become red hot while contacting the glass because it would stick to the glass, and at the same time, if the iron is too cool it will cause the glass to crack.

PAUL J. ALLEN.

Fort Worth, Texas.

Vapor Line Suspension

IN order to prevent undue vibration and to support long spans of vapor lines, Lion Oil Refining Company is using spring cushions at intervals instead of the usual solid supporting equipment. A frame is used which rests upon some solid part



Vapor Line Suspension

of the plant with a cross member above the line to be supported. Compression springs are used, equipped with eye bolts and stirrups. Straps are placed around the vapor lines to which the lower end of the spring is attached, while the upper end is fastened to the cross-member above them.

Water Drains in Boiler House

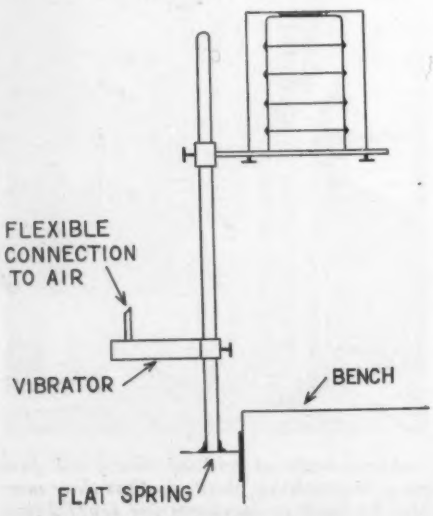
ONE OF the operators at Skelly Oil Company's Fairfax, Okla., No. 2, gasoline plant, while it was under construction last summer, suggested to the construction superintendent that an improvement could be secured in the boilerhouse, if the drains were installed so that water could be removed immediately, and without causing an accumulation of scale and mud on the floor. The idea consisted of

Boiler Room Drain Pipes

independent drain pipes placed in the floor of the building so they would extend outside and connect to a common sewer pipe beside the boilerhouse. A shallow depression was made in the concrete floor while it was being poured, so that water from each of the four boilers would flow through separate openings. When the lower hand hole plates of the oil field boilers are removed, sludge, scale and water runs directly to the opening in front of it, instead of pouring over the floor and beside the boiler. When the boiler is placed back on the line, it requires only a short time with a water hose to clean the floor thoroughly. The cost of the installation was small, but the returns are great in appearance and in labor saved.

Air Shaker for Sieve Test

THE cost of a mechanical shaking machine was saved by utilizing a second-hand air vibrator such as used for accelerating flow in a clay hopper.



Shaking Machine

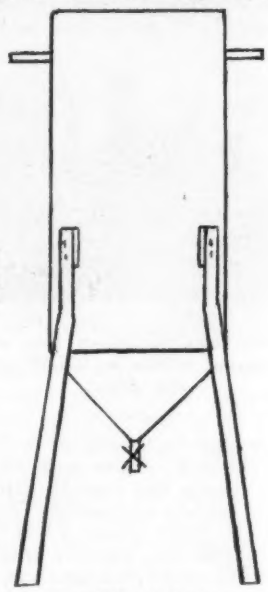
A half-inch rod was fastened vertically to the work bench through a flat spring. The vibrator was clamped near the bottom of this rod and the sieve-holder nearer the top. By this placement the vibrations at the lower end of the rod were multiplied by the distance between the clamps, thus producing sufficient motion to sieve the clay.

G.F.F.

Olean, N. Y.

Portable Agitator

THE frequent necessity for treating small lots of oil led to the use of the small agitator shown in the sketch. One- and five-gallon sizes were found convenient for processing those batches too small to be handled in the permanent lead-lined agitators of 15 to 20 gallon capacity.



Portable Agitator

They were fabricated from light-gauge black iron with the height approximately twice the diameter and a 45 degree cone. The apex of the cone was fitted with a large bore, quick-acting valve for rapid sludge dumping. The legs were made of angle iron toed-out somewhat to give greater stability. These legs were bolted to lugs welded to the shell so that they could be removed when occasion demanded. Welded handles were provided for easy carrying. GEO. F. FITZGERALD,

Olean, N. Y.

Sludge Light

AT NIGHT a lube oil acid treater has a difficult time determining the proper sludge particles because no matter how well lighted the top of the agitator, the sludge particles are not well defined by the reflected light. A device which solves this difficulty to complete satisfaction is very simply made and used as follows:

An ordinary large-headed flash light is take and its glass replaced with a disk of translucent opal glass. In use the agitator sample is placed on top of the opal glass, and the light shining up from beneath, illuminates each sludge particle with great definition, thereby making the treatment altogether independent of light conditions in his judgment of the treat.

Beaumont, Texas HARRY VINOCK